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### Citation Details

Anderson, Shelby L.; Tushingham, Shannon; and Buonasera, Tammy Y., "Aquatic Adaptations and the Adoption of Arctic Pottery Technology: Results of Residue Analysis" (2017). *Anthropology Faculty Publications and Presentations*. 125.  
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**AQUATIC ADAPTATIONS AND THE ADOPTION OF ARCTIC POTTERY  
TECHNOLOGY: RESULTS OF RESIDUE ANALYSIS**

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The late adoption of pottery technology in the North American Arctic between 2500 and 2800 years ago coincides with development of a specialized maritime economy. Arctic pottery technologies present an excellent case study for examining possible correlations between hunter-gatherer pottery and aquatic resource use. Review of the timing and distribution of early pottery in Alaska shows that early pottery is rare and dates at the earliest to 2500 years ago; earliest pottery is found in small numbers and primarily in coastal areas. Despite expectations that pottery use would be strongly linked to marine lipids, biomarkers and compound specific  $\delta^{13}\text{C}$  values of 20 sherds from the Cape Krusenstern site complex, dating from 2700 to 200 cal B.P. years ago, are most consistent with freshwater aquatic resources; mixtures of freshwater aquatic, marine aquatic, and terrestrial resources are also possible. While additional analysis of a larger sample and zooarchaeological reference specimens is necessary, our study suggests that the development of pottery production by Arctic peoples is more complex than previously appreciated. This research is the first synthesis in over 30 years of early pottery in Alaska and is also the first to include residue analysis of a small sample of pre-1500 B.P. pottery.

## **ADAPTACIONES ACUÁTICAS Y LA ADOPCIÓN DE LA TECNOLOGÍA ÁRTICA DE POTERÍA: RESULTADOS DEL ANÁLISIS DE RESIDUOS**

La adopción tardía de la tecnología cerámica en el Ártico Norteamericano entre 2500 y 2800 años atrás coincide con el desarrollo de una economía marítima especializada. Las tecnologías de cerámica del Ártico presentan un excelente estudio de caso para examinar las posibles correlaciones entre la cerámica cazador-recolector y el uso de los recursos acuáticos. La revisión del momento y de la distribución de la cerámica temprana en Alaska demuestra que la cerámica temprana es rara y comienza al más temprano hace 2500 años; la cerámica más antigua se encuentra en pequeñas cantidades y principalmente en las zonas costeras. A pesar de las expectativas de que el uso de cerámica estaría fuertemente ligado a los lípidos marinos, los biomarcadores y los valores del  $\delta^{13}\text{C}$  de compuestos específicos de 20 tiestos del complejo del sitio de Cape Krusenstern, que datan entre 2700 a 200 años calibrados antes del presente, son más consistentes con los recursos acuáticos de agua dulce; También son posibles mezclas de recursos acuáticos de agua dulce, acuática marina, y recursos terrestres. Mientras que el análisis adicional de una muestra más grande y de los especímenes de referencia zooarqueológica es necesario, nuestro estudio sugiere que el desarrollo de la producción de la cerámica por los pueblos árticos es más complejo que apreciado previamente. Esta investigación es la primera síntesis en más de 30 años de cerámica temprana en Alaska y también es la primera en incluir el análisis de residuos de una pequeña muestra de cerámica pre-1500 años antes del presente.

Archaeologists have put forth various hypotheses to explain the adoption of pottery technology by hunter-gatherer groups. These explanations include the efficiency of pottery vessels over other container technology, increased sedentism, population pressure and related increased needs for storage, and/or a change in food processing needs related to diet change. There are several examples from across the world of an association between pottery and increased use of aquatic resources. For example, recent residue analysis of lipids extracted from Incipient Jomon pottery dated to around 16,000 cal B.P., established that the predominant use for these early vessels was in the processing of aquatic resources (Craig et al. 2013; see also Lucquin et al. 2016). Early pottery in northern Colombia (Oyuela-Caycedo 1995) and interior Amazonia (Roosevelt 1995) are associated with use of estuarine resources such as fish and shellfish. Early pottery from southeastern areas of North America are found in coastal and riverine areas where shellfishing occurred, although the link between the two is not clear (Sassaman 1995). Alternatively, the adoption of pottery may be linked to the exchange, consumption or sharing of prestige foods and the associated development of social relationships (Hayden 1995; Taché and Craig 2015).

The late adoption of pottery technology in the North American Arctic, occurring sometime between 2800 and 2500 years ago, coincides with the development and spread of an increasingly specialized maritime economy. The Arctic environment is at the very fringe of where pottery making is possible; the moist and cold environment, along with a short season for pottery production and a lack of fuel, do not favor pottery production (Frink and Harry 2008; Harry and Frink 2009). Nevertheless, the abundance of pottery in post-1500 B.P. sites of the western Arctic indicates that pottery was an important part of the hunter-gatherer tool kit in this

region that may be linked to marine resource use (Farrell et al. 2014; Solazzo et al. 2008; Solazzo and Erhardt 2007). The age, distribution, and use of pre-1500 B.P. pottery technology in Alaska is, however, not well understood and presents an excellent case study for further examining possible correlations between hunter-gatherer adoption of pottery technology and aquatic resource use.

In this paper, we review the timing and distribution of early pottery in Alaska and explore the link between pottery adoption and aquatic resource use through lipid and compound specific stable isotope analysis (CSIA) of 20 pottery vessel fragments from the Cape Krusenstern site complex in northwest Alaska (Figure 1). This is the first North American Arctic pottery residue study to include samples that pre-date 1500 cal B.P.. Although our early (pre-1500 cal B.P.) pottery sample size is small (n=2), this analysis lays the groundwork for better understanding why pottery was adopted.

## **The Adoption of Pottery Technology in the North American Arctic**

### *Why do Hunter-Gatherers make Pottery?*

The specifics of how, when, and why hunter gatherers adopted pottery around the world is variable and dependent on regionally specific cultural and environmental contexts (see Jordan and Zvelebil 2009 for a recent overview). There are technological and economic advantages of

pottery over other container technology that could have led to the invention and spread of early pottery in hunter-gatherer groups. These include improved time management in both production and cooking (Crown and Wills 1995; Schiffer and Skibo 1987). Unlike fiber, wood, or skin containers, multiple ceramic vessels can be produced consecutively with little additional effort (Brown 1989; Eerkens et al. 2002). Foods that require prolonged soaking or cooking, (e.g., seeds), can be more efficiently processed in ceramic vessels (Arnold 1985) that can be both directly and indirectly heated. Most other container types cannot be directly heated (Rice 1999). Ceramic vessels can also hold liquids, withstand abrupt temperature changes (Brown 1989), and provide better long term storage for foods in most environmental contexts. These ceramic technological properties increase the range of available food that people can consume and save production and cooking time that can be used in other pursuits (Arnold 1985; Hoopes and Barnett 1995:5; Rice 1999). It is possible that in some cases increased diet breadth leads to innovation or adoption of new ceramic technologies (Rice 1999). Furthermore, caloric returns from the incorporation of lower ranked resources such as small fish and shellfish into the diet could be improved by prolonged cooking made possible by ceramic technology. Alternatively, the relationship between aquatic resource use and pottery could be coincidental. Pottery production and use is more feasible as hunter-gatherers become sedentary, which can occur when people focus on predictable and abundant resources such as aquatic resources. Or, early pottery use could have emerged for ritual use or in social contexts that included feasting and other forms of increased social interaction or exchange that may have included aquatic resources (Hayden 1995; Taché and Craig 2015; Harry et al. 2009). This would explain relatively small early pottery sample sizes observed in most contexts and the appearance in some cases of non-vessel ceramic forms prior to pottery production.

## *The Origins and Timing of Pottery Adoption in Alaska*

The study of pottery from the North American Arctic provides an opportunity to research these processes of adoption in an area where pre-contact pottery use is poorly understood. In the North American Arctic, the adoption of pottery coincides with increased residential sedentism and an increasing reliance on marine resources along the coasts of the Bering Strait and northwest Alaska. This is also a period of increased interaction across the Bering Strait; pottery is one of several artifact types that appear for the first time in the Alaskan record sometime between 2750 and 2450 years ago, during the Choris phase. The origins of North American Arctic pottery are generally accepted to lie to the west, in the Chukchi Peninsula and surrounding regions of Chukotka (Figure 1) (Ackerman 1982; Dumond and Bland 1995) where it appears somewhat earlier, perhaps around 5000 ya (see Ackerman 1982; Dumond and Bland 1995 for more discussion)<sup>i</sup>. Although there are a few pottery fragments possibly associated with earlier Denbigh or Arctic Small Tool Tradition (4500-2800 B.P.) components at the Engigstciak, Punyik Point, and Coffin sites in northern Alaska (Figure 2) (Ackerman 1982: 14; Stanford 1971, 1976:16; Stimmell 1994), the Choris phase is more widely accepted as the first adoption of ceramic technology in Alaska. After 2300 years ago Norton phase (known as Near-Ipiutak in northern Alaska) pottery types are found at a small number of sites across a wider area of western Alaska (Figure 2). Beginning around 1500 years ago and increasingly after 1000 years ago, early pottery types were replaced across the North American Arctic by a significantly different pottery tradition associated with Birnirk and Thule cultures. This tradition spread with



the ancestors of modern Iñupiat people across northern Alaska into the central Canadian Arctic and south into ancestral Yup'ik, Cup'ik, and Alutiiq (Sugpiaq) regions of southwest Alaska.

### *Alaskan Pottery Traditions*

There are two major pottery traditions in northern Alaska: Pre-1500 B.P. pottery associated with Choris, Norton (or Near Ipiutak) Phases, and post-1500 B.P. pottery associated with Birnirk, Thule, and other late pre-contact cultures. In northern Alaska, pre-1500 B.P. Choris and early Norton or Near Ipiutak pottery was typically thin-walled and decorated with cord marking or with linear or check stamping. Northern Alaska vessels had a globular vessel shape (e.g. Giddings and Anderson 1986), while vessels from southwest Alaska had a more cylindrical or barrel shape (e.g. Dumond 1981). Size estimates are not possible for the earliest pottery vessels due to the small number of available samples. Post-1500 B.P. pottery was thicker and frequently undecorated or decorated in a variety of regional styles. Vessels were flat bottomed and cylindrical or flower pot-like in shape. Vessel size was typically on the smaller side, between 25 and 50 cm diameter (Anderson 2011:92, see also Frink and Harry 2008 for ethnographic examples), but some regional variation in size is likely given that size estimates have not been made for many assemblages. Temper type varied from region to region throughout time. The shift from thin-walled, globular shaped vessels to thick-walled flat bottomed vessels after 1500 B.P. suggests a change in the way pottery was used to process foods; a shift from direct to indirect cooking is possible. However, charring is common on vessel exteriors in post-1500 B.P. pottery (Anderson 2011), suggesting that later vessels were sometimes placed directly in the cooking fire rather than exclusively indirectly heated. There is

a significant shift in pottery abundance after about 1500 years ago in northern Alaska, with pottery found at most coastal or coastal margin sites dating to after 1500 years ago. This is likely partially a function of preservation and an overall increase in known sites that post-date 1500 B.P., but may also reflect a change in pottery use or importance. At least some coastal peoples in northern Alaska briefly abandoned pottery technology between about 1750 and 1150 years ago, during the Ipiutak phase (see Mason 2006; 1998 for discussion); a satisfactory explanation for this phenomenon has not yet been offered.

### *The Role of Pottery in Pre-Contact Northern Alaskan Cultures*

The timing and distribution of early pottery technology in Alaska suggests a possible link between an expansion in maritime adaptations and early pottery use that began around 2700 B.P.. Was the adoption of pottery in northern Alaska associated with an expanding diet breadth and/or related to an increasingly marine focused diet? The relationship between expanding diet breadth and pottery is most common in the use of r-selected (e.g. fruits, seeds, shellfish) rather than k-selected species (e.g. seals, walrus) (Hoopes 1995; Rice 1999) that are associated with marine resource use during this period in northern Alaska. However, fish and marine mammal fat rendering may have been a particularly important form of processing that pottery technology facilitated in Arctic settings, given the critical nutritional role of fats in plant and carbohydrate poor northern climates (Fitzhugh 2003:68-70). Marine mammal and fish oil was both a food unto itself and an important component in the storage process; dried and otherwise processed meat and plant products were often preserved in oil for later consumption (e.g., Burch 1998:147,

189). Oil was also a crucial source of fuel for light and heat, particularly where wood was sparse (Burch 1998:244).

Pottery vessels may have been particularly suited to processing fish and mammal bone and fats to extract added nutrients and fats since prolonged boiling is possible in ceramic vessels but not in other types of containers used in the Arctic such as baskets or skin bags. Harry and Frink (2009:334) argue that while some Northern peoples did boil fat to render oil, in western Alaska it was more common to put blubber in seal pokes and bury it underground where the fat would render itself (see also Spray 2002). Instead, they hypothesize that cooking vessels were adopted in this region because of culinary preferences for parboiled foods. Fish oil, however, would have been difficult to render in seal pokes since fish have more dispersed and sparser deposits of body fat than marine mammals and would not have self-rendered in the same way as marine mammal fats. Beluga fat also had to be cooked to transform it into oil; self-rendering did not work (Burch 1998:165). On the Selawik and Kobuk rivers of northwest Alaska, there are 19<sup>th</sup>- 20<sup>th</sup> century accounts of people processing fish for oil by boiling them indirectly in a large wooden pot (Burch 1998:146). After boiling, the water was left to cool and the fat rose to the top. Similar processing in ceramic vessels could have taken place. Russian reports of Kodiak Island people melting whale fat in clay vessels (de Laguna 1939) further support the idea that post-1500 B.P. ceramic cooking vessels were used for oil rendering at least occasionally.

Alternatively, perhaps pottery was adopted for social reasons, possibly related to increased interaction across the Bering Strait. In a study of early northeastern North American pottery use, Taché and Craig (2015) find that pottery was used for storing or processing

exchange commodities, such as fish oil. They argue that the development of pottery technology was related to social developments in hunter-gatherer society, such as feasting and social relationships. Such processes are entirely possible in Alaska; in the late pre-contact and contact era marine mammal (and possibly fish) oil was an important exchange item that could have been processed and/or transported in pottery containers (Burch 2005). Or, pottery vessels could have been used in feasting contexts. The small number, all decorated, of early pottery in Alaska (Ackerman 1982) provides tentative support for this hypothesis.

### **A Revised Synthesis of Early Alaskan Pottery**

While the general pattern of pottery adoption in Alaska is fairly well understood, data on early pottery is limited and largely based on a review of the evidence for early pottery in Alaska and the Russian Far East completed in the early 1980s (Ackerman 1982). In order to better understand the timing and distribution of pre-1500 B.P. pottery in Alaska, we undertook a review and synthesis of post-1980 published and unpublished literature and site data in an expansion of the prior review. This involved an examination of Alaska state site records for site documents and associated reports and records that contained information about early pottery sites and any sites reported to contain precontact pottery or ceramic material. Out of more than 45,000 site records and 15,000 reports in the Alaska state site database, we identified early site records including 41 associated with the Choris phase and 100 records associated with the Norton phase, as well as 103 site records that mentioned precontact ceramics, and 179 site records that reported

precontact pottery. We reviewed the site forms, associated reports, and other regional gray literature published since 1980 (e.g., Anderson 1988; BIA ANCSA 1997; Bundy 2007; Giddings and Anderson 1986; Schaaf 1988; Tremayne 2014; see Table 1 for additional references) and compiled detailed information about site age, cultural affiliation, and ceramic materials. Within this sample, we compiled a database of 47 early pottery sites (Table 1), which include sites that met one or more of the following criteria:

- Pottery-bearing sites dating to before 2000 B.P.
- Sites identified as dating to the Denbigh (or Arctic Small Tool Tradition), Choris, or early Norton (e.g. Smelt Creek Phase) phases
- Presence of early pottery types (Cord marked, Check stamp, Linear stamp, Textile impressed, Diamond stamp)

### *Early Pottery Distributional Patterns*

Significantly, our findings revealed patterns in the distribution of early Alaskan pottery that are consistent with those observed by Ackerman over 30 years ago. A large amount of archaeological work has taken place in Alaska over the past three decades but very little additional early Alaskan pottery was recovered (Table 1). As a result, we conclude that early Alaskan pottery is relatively rare and generally found in small numbers at only a few sites. For example, in a recent regional-scale analysis of 8,393 pottery sherds from northwest Alaska (Anderson 2016; Anderson et al. 2016; Anderson et al. 2016), only 9 early pottery samples were identified.

Our synthesis revealed interesting distributional patterning of early pottery sites. First, early pottery sites are primarily located in coastal areas (Figure 2). While it is true that research is biased towards coastal areas in northern and western Alaska, several significant projects have taken place in interior regions of northwest Alaska since 1980 and the interior sample of early pottery is still very small. Second, while we identified a few early pottery sites dating to as early as 2500-2600 B.P. (e.g. Cape Espenberg, Choris, Iyatayet, and several Nunivak Island sites), the majority of early pottery sites date *after* 2300 B.P. (Table 1). Third, the earliest sites (2500 to 2300 B.P.) are distributed from northern Alaska to as far south as Nunivak Island, with slightly later (after 2300 B.P. to 2000 B.P.) sites found over a wider area of the Yukon-Kuskokwim delta and southwest Alaska. Finally, Norton pottery is widespread in southwest Alaska after 2000 B.P..

Unfortunately, the earliest pottery sites remain poorly dated. They are often from sites or contexts that are minimally dated and often by outdated radiocarbon methods (e.g., solid carbon dating). Dating of Choris and early Norton phase sites is often based on relative dates from pottery types rather than absolute dates on associated site materials so it remains unclear when the pottery technology first appeared in Alaska; a total of 24 out of the 47 early pottery sites lack absolute dates (Table 1). In numerous cases the earliest pottery was recovered from uncertain contexts (e.g., at Choris and Iyatayet).

To summarize, review of data on early pottery sites in Alaska reinforces that there is a link between early pottery use and coastal occupation. Residue studies (Farrell et al. 2014;

Solazzo and Erhardt 2007; Solazzo et al. 2008) of both pots and lamps from Alaska indicate a link between post-1500 B.P. northern Alaskan pottery and marine resource use. However, until now, there was no residue data on pre-1500 B.P. pottery to address the question of changing use over time<sup>ii</sup>. Prior studies also did not incorporate CSIA of fatty acids, which can distinguish freshwater and marine signals. We undertook residue analysis to further explore pottery use in the Arctic, with a particular interest in possible links between pottery and aquatic resource use. We examined residue data from a data set that spans a longer temporal period than prior studies, encompassing the shift in pottery traditions before and after 1500 B.P.. Questions we seek to address here are: 1) What types of resources were people processing in pottery vessels over the last 2500 years? and 2) Did resource use change over time? Our overall sample size is relatively small, particularly the pre-1500 cal B.P. pottery sample (n=2), and as a result we consider this analysis exploratory.

### **Residue Analysis Sample Selection and Methods**

Pottery samples for residue analysis were selected from several different sites at the Cape Krusenstern site complex, located in northwest Alaska. The site complex encompasses a 4200-year record of past human coastal occupation (Anderson and Freeburg 2013, 2014). The majority of sites at the complex that date before 2000 B.P. come from short term occupations that typically consist of a scatter of surface artifacts and occasional hearth features. After 2000 B.P., the prevalence of semi-subterranean occupation sites and settlements points to an

increasingly settled lifeway and an increase in local population over time. Beginning around 500 years ago, there is a shift in regional and local settlement patterns, with a decrease in settlement size and a relocation of settlements to previously unoccupied areas of the site complex (see Anderson and Freeburg 2013, 2014 for more details).

Twenty pottery samples were selected from several types of sites including surface scatters, semi-subterranean occupation features, and indeterminate features that could be occupation locations or storage features. Sample ages range from approximately 2700 to 200 cal B.P., with the majority of the samples dating to after 1000 cal B.P. (Table 2). Dates were obtained on associated materials, which were selected from the levels and units as closely associated with the ceramic sample as possible given available datable material (see Table 2). In the future it may be possible to directly date food crusts associated with pottery and, perhaps, directly date the last uses of particular pots (e.g., see Heron and Craig 2015). In the current study, however, dating of charred residues from ceramic sherds was avoided due to uncertainties arising from unknown and heterogeneous organic inputs to charred material.

Samples are all from distinct vessels and were fragmentary when recovered. All of the available samples were undecorated vessel body sherds; rim sherds and decorated sherds were not available from dated contexts. Thickness, temper, and other technological characteristics were taken into consideration when selecting samples to avoid sampling the same vessel twice from the same context. Several of the sherds had interior and/or exterior surface residues that were apparent without magnification (Table 2). Details of laboratory methods including extractions, derivatizations and instrumental parameters are provided in supplemental materials



(See Supplemental Text 1). A brief summary of procedures used to extract and analyze lipids from the 20 pottery sherds follows.

Lipids were extracted from powdered sherds with solvents (chloroform and methanol) and sonication, derivatized to methyl esters, and then analyzed for overall composition as well as the  $\delta^{13}\text{C}$  values of individual fatty acids. Only absorbed residues were analyzed. Compositional analysis of sherd lipids was performed at the Gang Laboratory (Laboratory for Cellular Metabolism and Engineering) at Washington State University in Pullman Washington using GC/TOF-MS. Compound-specific  $\delta^{13}\text{C}$  analysis (CSIA) of individual fatty acids, using GC combustion isotope ratio mass spectrometry (GC-C-IRMS), was performed at the UC Davis Stable Isotope Facility in Davis, California.

### **Criteria Used to Interpret Lipid Sources**

Recent experimental and archaeological work has made it possible to identify aquatic lipids in archaeological contexts (Copley et al. 2004; Craig et al. 2011; Evershed et al. 2008; Hansel et al. 2004; Heron et al. 2010, 2013). Widely-accepted biomarker criteria for heating aquatic lipids in pottery vessels currently includes a combination of at least one of three isoprenoid fatty acids—4,8,12-trimethyltridecanoic acid (4,8,12-TMTD), 2,6,10,14-tetramethylpentadecanoic acid (pristanic acid), and 3,7,11,15-tetramethylhexadecanoic acid (phytanic acid)—together with,  $\omega$ -(*o*-alkylphenyl)alkanoic acids of at least 18 and 20 (and

preferably, also 22) carbons (Evershed et al. 2008). Combining aquatic biomarkers with CSIA can provide strong evidence for the processing of marine fauna (Buonasera et al. 2015; Copley et al. 2004); alternatively, it can suggest freshwater aquatic contributions, or a mixture of terrestrial and aquatic resources (Craig et al. 2007, 2011; Taché and Craig 2015; Lucquin et al. 2016).

### *Biomarker Compounds Used to Identify Aquatic Resources*

Isoprenoid fatty acids 4,8,12-trimethyltridecanoic acid (4,8,12-TMTD) 2,6,10,14-tetramethylpentadecanoic acid (pristanic acid), and 3,7,11,15-tetramethylhexadecanoic acid (phytanic acid) are present in aquatic animal fats; they are rarely encountered, and in only very low amounts, in terrestrial mammals, and are not present in plant oils (Ackman and Hooper 1968; Ackman 1989; Copley et al. 2004; Evershed et al. 2008). Phytol, present in the chlorophyll of photosynthesizing organisms, is biologically modified to 4,8,12,-TMTD, phytanic acid, and pristanic acid as it moves through aquatic food webs (Ackman 1989:23). The presence of one or more of these isoprenoid fatty acids is used to detect the processing of aquatic products in archaeological pottery and features (Copley et al. 2004; Craig et al. 2007; Cramp et al. 2014; Farrell et al. 2014; Hansel et al. 2004; Heron et al. 2010).

In addition to isoprenoid fatty acids, the presence of  $\omega$ -(*o*-alkylphenyl)alkanoic acids with 18, 20, and 22 carbons provides evidence that aquatic products were processed in the presence of heat. Experiments indicate that these compounds form when tri-unsaturated fatty acids like 18:3, 20:3, and 22:3 are exposed to temperatures above 270°C in an anoxic environment (Evershed et al., 2008:105). Unlike terrestrial mammal fats, aquatic fats/oils have high amounts of polyunsaturated fatty acids that are 20 and 22 carbons long. Heating these fats

in pottery vessels produces  $\omega$ -(o-alkylphenyl)alkanoic acids 20 and 22 carbons long (Evershed et al., 2008).

Detection of  $\alpha,\omega$ -dicarboxylic acids (sometimes referred to *diacids*) can provide further evidence that substantial amounts of unsaturated fatty acids were once present in a residue (Buonasera 2013; Passi et al. 1993; Regert et al. 1998). These compounds are formed from the oxidation of C-C double bonds, and their length may be representative of double bond positions in the original unsaturated fatty acids (Evershed et al. 2008; Passi et al. 1993). Evershed et al. (2008:106) found that  $\alpha,\omega$ -dicarboxylic acids between C8 and C11 long were formed “in appreciable amounts” during experimental heating of marine oils.

#### *Compound Specific $\delta^{13}\text{C}$ Analysis (CSIA)*

Application of CSIA to ancient lipids compares  $\delta^{13}\text{C}$  16:0 and  $\delta^{13}\text{C}$  18:0 values to those from modern reference fats that have been adjusted to account for contributions of industrial carbon (Craig et al. 2011:17914; Regert 2011:196). Palmitic ( $\text{C}_{16:0}$ ) and stearic ( $\text{C}_{18:0}$ ) acids are used in these comparisons because they are typically the most abundant lipids encountered in ancient organic residues. Marine animal fats are significantly more enriched in  $^{13}\text{C}$  than terrestrial animal fats, allowing for discrimination between these resources (Copley et al. 2004; Cramp et al. 2014; Craig et al. 2007; Craig et al., 2011; Choy et al. 2016). Freshwater fish and salmonids (including freshwater and anadromous species) overlap with non-ruminant animal fats, but not with wild ruminant animal fats (Taché and Craig 2015; Craig 2007; Craig et al. 2011). Even though anadromous salmon do not eat once they enter freshwater systems,  $^{13}\text{C}$  values for reference fats from these species are less enriched than fats for many marine

organisms and at the upper (more enriched) end of freshwater fish (Choy et al. 2016; Taché and Craig 2015).

In the current study, we identified the heating of aquatic resources in pottery vessels using a variety of data, including, at a minimum, the presence of one or more isoprenoid fatty acids (4,8,12-TMTD, phytanic acid, or pristanic acid), as well as  $\omega$ -(*o*-alkylphenyl)alkanoic acids 18 and 20 carbons long. Further information on the sources of sherd lipids was provided by  $\delta^{13}\text{C}$  values for  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$ .

Other compounds that supported aquatic resource designations included long chain saturated and unsaturated fatty acids with 20 and 22 carbons, or more, and ratios of palmitic ( $\text{C}_{16:0}$ ) to stearic acids ( $\text{C}_{18:0}$ ) greater than 1 (Heron et al. 2010; Taché and Craig 2015). Fatty acids longer than 18 carbons, especially unsaturated fatty acids longer than 18 carbons, are rare in terrestrial animal fats, but abundant in aquatic resources (Evershed et al., 2008). They are also found in some plant seed oils and are components of the cuticular waxes of terrestrial plant leaves (Gunstone 1999). Given the local environment, however, large systematic contributions from terrestrial plant sources seem less likely than contributions from aquatic organisms. We also noted the presence of  $\alpha,\omega$ -dicarboxylic acids ranging from carbon chain lengths of C7-C12.

## **Residue Analysis Results**

### *Aquatic Biomarkers*

Most sherds have combinations of markers that are highly suggestive of aquatic resources-- though not all are definitive according to accepted criteria. As detailed in Table 3, six samples contained one or more isoprenoid fatty acids, 11 samples contained APAAs, most samples had saturated and monounsaturated fatty acids 20 and 22 carbons long, and most also had ratios of palmitic acid to stearic acid greater than one ( $>1$ ). Additionally,  $\delta^{13}\text{C}$  values for  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  are consistent with aquatic organisms.

Two samples (14110 and 15146) have ratios of palmitic to stearic acid that are much lower than one ( $< 1$ ), which could suggest a substantial terrestrial animal (especially ruminant) contribution. On the other hand, 14110 had some of the more enriched  $\delta^{13}\text{C}$  values in this study. The  $\delta^{13}\text{C}$  values of 14110 are more consistent with aquatic contributions, and especially salmonids, than with ruminants. Both sherds (14110 and 15146) also have less abundant and less complex lipid contents than many other sherds, which could indicate differences in use or less favorable preservation conditions.

The strongest candidates for processing of aquatic resources were samples 15151, 14514c, and 14515b. These three sherds each exceeded the minimum acceptable criteria for aquatic resources and retained two or three isoprenoid fatty acids, APAAs 20 carbons long, and saturated and monounsaturated fatty acids 20 and 22 carbons long. These sherds also contained abundant degradation products of unsaturated fatty acids (dicarboxylic fatty acids and dihydroxy fatty acids) and high palmitic to stearic acid ratios. Figure 3 shows a total ion count chromatogram (TIC) for 14515b. All three samples were collected from house features at the Late Western Thule site (see Giddings and Anderson 1986 for more site information). Unfortunately, no contextual information is available for these samples, which were collected from disturbed contexts at these previously excavated features. There are no other specimens from these

features available for analysis as previously excavated ceramic specimens from this site were lost at sea during post-excavation transport (see Giddings and Anderson 1986). Regardless, contextual information from excavations were limited to the house level.

### *Compound specific $\delta^{13}\text{C}$ values*

Stable carbon isotope values of sherd lipids are consistent with an aquatic origin, but do not indicate a primarily marine origin. More specifically,  $\delta^{13}\text{C}$  values for  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  place sherd lipids most convincingly in the range of freshwater or anadromous fish (Figure 4 and Table 3)(Supplemental Figure 1 and Supplemental Table 1). These values partially overlap reference fats for wild non-ruminant terrestrial mammals though they do not overlap wild ruminant reference fats (see discussion below). It is also possible that the sherd  $\delta^{13}\text{C}$  values could result from mixtures of marine and freshwater resources, or aquatic and terrestrial resources.

As noted above, comparison of  $\delta^{13}\text{C}$  values for  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  in sherd lipids argues against a predominantly ruminant origin. Among ruminants, differences in the biosynthetic origins of  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  produce a pattern where  $\text{C}_{18:0}$  is 1-6 ppm more depleted in  $^{13}\text{C}$  than  $\text{C}_{16:0}$  (Evershed et al. 2002:81; Taché and Craig 2015:184). In contrast to the pattern observed in ruminant fats, the  $\delta^{13}\text{C}$  values in our samples show an opposite relationship with  $^{13}\text{C}$  more depleted in  $\text{C}_{16:0}$  than in  $\text{C}_{18:0}$  (Figure 4) (Supplemental Table 1).

To summarize, the  $\delta^{13}\text{C}$  values for  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  do not indicate that sherd lipids were derived from primarily marine or primarily ruminant sources. Based on our CSIA results, sherd lipids could arise from processing: 1) primarily freshwater fish, 2) primarily non-ruminant terrestrial mammals, or 3) mixtures of various resources. When this isotopic information is

combined with data from lipid compositional analysis, however, it supports an aquatic contribution rather than one dominated by non-ruminant terrestrial animals. Lipid compositional analysis shows a widespread presence of numerous aquatic biomarkers including APAAs 18 to 20 carbons long, isoprenoid fatty acids, and abundant monounsaturated and saturated fatty acids 20 and 22 carbons in length. Although not all of these compounds are always present in the same sherd, most sherds contain one or more aquatic biomarkers (Table 3). Further, if either marine resources or ruminants were routinely processed in pottery vessels, we might expect a larger sample to include many sherds with remnant fats that plot closer to, or within, the marine or wild ruminant zones.

## **Discussion**

### *The Origin of Lipids in Pottery: Potential Confounding Factors*

It is important to consider factors that may confound our results, specifically the possibility that the identified residues are not associated with the cooking and preparation of certain foods *per se*, but rather may relate to pottery production and maintenance. For example, post-contact Native Alaskans incorporated dog hair and excrement, seal blood and seal oil into the clay itself during clay preparation (Arnold and Stimmell 1983; Fienup-Riordan 2005). During the late 19<sup>th</sup>/early 20<sup>th</sup> century there are various reports of treating unfired vessels with seal oil (Fienup-Riordan 1975:14; Oswalt 1952:20), burnt fish eggs (VanStone 1989:28), and

potentially other animal oils or blood. In some cases pottery vessels were treated or seasoned after firing by placing a “broth” of water and fish backbones into the vessel and cooking it all day to impart a permanent fishy flavor to the vessel (Oswalt 1952). Vessels were sometimes smeared with blood or oil after firing, and were sometimes re-oiled after a period of use (Reid 1989:171). Vessels were also reportedly lined with skins or membrane material once finished, likely to reduce vessel permeability (Arnold and Stimmell 1983; Reid 1989:171).

While materials added to the vessel prior to firing likely broke down during the firing process, the effect of post-firing treatment with oils and fats on the residue results is unclear. Surface residues were removed during sample pre-treatment, but absorbed residues from pottery maintenance rather than use could be included in analytical results. Mixing of residues related to maintenance versus residues associated with use could result in signals like those encountered in this study. Future experimental studies would help elucidate such processes. This issue could also be addressed in future studies by comparing bulk  $\delta^{15}\text{N}$  and compound specific  $\delta^{13}\text{C}$  values between charred surface encrustations from burned food or other materials processed in the pots and residues absorbed in the ceramic matrix. Charred surface crusts can be complex mixtures of various biomolecular compounds, potentially from different tissues and different organisms, in addition to environmentally absorbed compounds. Several studies, however, suggest that combining bulk  $\delta^{15}\text{N}$  values of charred surface crusts with  $\delta^{13}\text{C}$  values of specific fatty acids ( $\text{C}_{16:0}$  and  $\text{C}_{18:0}$ ) extracted from the crusts can help to distinguish between freshwater, marine, and terrestrial sources (Craig et al. 2007; Heron and Craig 2015). Such data could prove especially useful for detecting systematic differences between absorbed versus surface residues in large samples of sherds.



### *Northern Alaskan Vessel Use*

The residue results are somewhat surprising in that the evidence does not indicate heavy or exclusive marine resource processing in ceramic vessels. These results differ from previous regional residue studies that identified marine residues and proteins in northern pottery samples (Solazzo et al. 2008; Solazzo and Erhardt 2007) or aquatic residues but not specifically marine versus freshwater residues (Farrell et al. 2014). This may be due to methodological differences. For example, Farrell et al. (2014) used GC and GC/MS but did not use CSIA. Their analysis, therefore, relied only on biomarkers as no isotope data were available. At present, the only way to separate freshwater and marine resources is through isotopic analysis since both resource types have the same suite of biomarkers. Our residue results also differ from our expectations for local diet and dietary change based on the results of on-going faunal analysis from Cape Krusenstern. Faunal research indicates a reliance on a variety of seal species. Caribou (the predominant local ruminant resource) are found in small proportions in faunal assemblages from across the site complex and fish remains were recovered in abundance from only a few sites, primarily dating to the late pre-contact period (approx. 500-250 cal B.P.) (Freeburg, personal communication 2016). Artifacts recovered from the site complex further indicate a reliance on marine resources, with fish associated artifacts (small net weights, smaller barbed hooks and gaff parts) more abundant in the late pre-contact assemblages (Freeburg and Anderson 2012; Giddings and Anderson 1986). The recovery of fish bone is likely partially impacted by preservation conditions at older sites and lack of small screen size use by previous investigators. Ethnographic evidence indicates that in addition to spring sealing activities at Cape Krusenstern,

people fished for several species of whitefish and other locally abundant fishes (e.g. char, grayling) in local lagoons and rivers (Burch 1998; Uhl and Uhl 1977). Interestingly, fishing was primarily a women's activity during the ethnographic period (Burch 1998), as was pottery making and use (Harry and Frink 2009). The predominant method of catching whitefish reported at Cape Krusenstern and in the surrounding area was through the construction of gravel and wood catchments at shallow lagoon mouths (Burch 1998:145; Uhl and Uhl 1977:11). After minimal construction, these structures would trap fish as water levels dropped with the outgoing tide. Such structures would not leave a trace in the archaeological record. Our residue results, therefore, provide a unique line of evidence about local diet in the absence of fish bone preservation and preservation of fishing related artifacts and structures.

### **Conclusions: Alaskan Adoption of Pottery and Aquatic Resource Use**

While much remains to be learned, our results indicate a more complex and interesting pattern of resource processing in ceramic vessels than anticipated at the outset of our study. The potential for a link between aquatic resource use and pottery use remains, although the evidence is strongest for post-1500 B.P. samples rather than for early, pre-1500 B.P. samples, which are small in number. Our synthesis of published and unpublished Alaskan ceramic data shows that few early Alaskan pottery sites date to before 2300 years ago; there are no dated pottery sites earlier than 2500 cal B.P.. Early sites are rare and are known primarily from coastal contexts. This research adds a sample and associated radiocarbon date to the early pottery data set, but the

known sample size for pre-1500 B.P. pottery remains quite small and residue data from early pottery is minimal. The residue evidence for both early and late pottery suggest either the use of primarily freshwater fish, or mixtures of various resources which could include marine resources. Our results do not suggest exclusive or predominantly marine resource use, contrary to our expectations at the outset of the study. Residue data also provides information about pre-1700 cal B.P. resource use that was not previously available as faunal and artifactual data from the site complex dating to before 1700 cal B.P. are very limited; the pre-1700 cal B.P. sample size, however, must be expanded to further explore pre-1700 cal B.P. diet.

Possible links between northern pottery use and expanding diet breadth or intensification remain. Our data does suggest that this was more likely after 1500 B.P. when shifts in pottery technology and abundance coincide with other evidence of growing population, sedentism, and reliance on marine and aquatic resources around western Alaska and the Bering Strait region (Anderson et al. 2016). Our pre-1500 cal B.P. sample size is too small to assess whether or not this was the case during the period of pottery adoption in Alaska. Perhaps pottery use met a need for resource intensification to support a growing and increasingly sedentary population at this time. Earlier use of pottery was possibly associated with other activities, such as social exchange or interaction during a period of rapid change in Alaska beginning around 2700 B.P.. The small amount of early pottery in Alaska could be a function of preservation or the small number of known sites from this time. As previously observed by Ackerman (1982) early pottery may have been prestige objects rather than everyday household items as seems to be the case after 1500 B.P. in later periods. That all of the known early pottery is decorated is interesting in regard to the latter hypothesis; prestige or ceremonial use is partially about display of pottery in social

contexts (Hayden 1995) and decorative elements may have been particularly important in these contexts. While oils were often rendered and stored in seal pokes and other animal organs, it is possible that storage and transport of the same commodities in small “curated” pottery containers increased their social impact and prestige value in exchange and social interactions.

Geochemical sourcing of a small number of early pottery shows overlap in source use between early and later ceramics in northwest Alaska, indicating that early pottery was made locally rather than imported from elsewhere (Anderson et al. 2011, 2016; Anderson 2016). Sourcing indicates that the pottery itself was made in the region; pottery may have been used to process or transport oils or other exchange items as a part of crucial social interactions that are well documented during the late pre-contact and contact eras in Alaska.

So why was pottery technology adopted in the North American Arctic around 2500 B.P.? Additional residue analysis, particularly of pre-1500 B.P. pottery, is needed to further evaluate this question. With additional samples, we may be able to use statistical methods to detect increased or decreased processing of aquatic resources over time. For example, Taché and Craig compared the relative use of marine resources between inland and coastal locations by applying a non-parametric statistical test to bulk C and N stable isotope results (2015:180). Future study should include a large reference sample of local native species to better characterize the range of variability in compound specific  $\delta^{13}\text{C}$  values; this is particularly important for northern Alaskan resources as much of the current CSIA reference data come from Europe or Asia. Comparison of pottery data to analysis of extractions from identified zooarchaeological specimens would also be informative. Comparison of early and late coastal pottery residue data with residue analysis of samples from interior northwest Alaskan sites would further inform on the question of pottery

use, as would experimental work to explore the relationship between animal products used in pottery production versus use, and the resulting residue signals.

Our study provides provocative results suggesting a longstanding association of pottery with aquatic resources over an interval spanning the initial adoption of pottery around 2500 – an interval when pottery was a novel technology and possibly a marker of social status—followed by an expansion in the use of pottery throughout Alaska around 1500 B.P.. Our data on early, pre-1500 B.P. pottery use, is quite limited (n=2) and these initial results need additional investigation. Later use of pottery may have provided a technological solution to changes in adaptive strategies occurring at this time, namely decreased group mobility, an expansion of maritime adaptations, and resource intensification. Our results, while far from final, suggest stability in association with aquatic resources, and contributes new data to the larger discussion about hunter-gatherer pottery and aquatic resource use in northern contexts.

*Acknowledgements:* This research was funded by the National Park Service through cooperative agreement (J8W07070032) managed by the Pacific Northwest Cooperative Ecosystem Study Unit. Many thanks to Eileen Devinney (NPS) for supporting residue analysis of Cape Krusenstern pottery samples and to Justin Junge for his assistance with Figure 2. Johonna Shea provided further assistance with preparation of graphics. Mary Soots provided the Spanish abstract. Permission for destructive analysis was obtained from the National Park Service prior to conducting residue analysis. No conflicts of interest were identified in a review of author financial interests and affiliations associated with this submission. We also thank Jelmer Eerkens

(UC Davis Anthropology), David Gang and Anna Berem (Laboratory for Cellular Metabolism and Engineering at WSU), and Chris Yarnes (UC Davis Stable Isotope Facility).

*Data Availability:* Electronic data associated with residue and isotopic analysis are stored at the University of Arizona School of Anthropology. Remnant ceramic specimens are stored at Portland State University Department of Anthropology.

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## Notes

<sup>1</sup> Thin-walled, linear stamp, cord marked, and textile-impressed pottery types are found in the region at sites associated with the Ust' Bel'skaia culture (3450 to 2450 years ago)(Ackerman 1982; Ponkratova 2006), while check stamped types are known from the slightly later but temporally and geographically overlapping Northern Chukotkan Late Neolithic culture beginning around 3000 years ago (Ackerman 1982:20). The roots of these early Chukotkan ceramic traditions are thought to be further afield, probably linked to Syalakh, Bel'kachinsk, and Y'myiakhtakh ceramic traditions that spread from the Yakutia region eastward to Chukotka and possibly into Alaska. The Alaskan Arctic Small Tool tradition could be an aceramic variant of Bel'kachinsk culture (Powers and Jordan 1990:268) while Ymyyakhtakh (approx. 4200 to 2500 ya) ceramics are thought to be the antecedent of Norton check stamped pottery that appears in Alaska perhaps as early as 2500 years ago (Dumond and Bland 1995). Post-1500 B.P. ceramics found at coastal Chukotkan sites are the same as those found on the Alaskan coasts of the Bering Strait at this time (Ackerman 1982; Ponkratova 2006); pre-1500 B.P. coastal Chukotkan ceramics have not been identified. Ceramic materials are found to the south on the Okhotsk Sea coast during the latter part of the Tokareva period, beginning perhaps as early as 3500 years ago (Ponkratova 2006:133-134). Much earlier ceramic traditions are known from the Primor'e and Priamur'e of the southern Russian Far East, beginning around 13,000 years ago (Figure 1)(Zhushchikhovskaya 2006, 2009, 2010; Zhushchikhovskaya and Shubina 2006), but clear links between this earlier tradition and those of the Bering Strait region have not been established.

<sup>2</sup> Although note that dates or age information is not available for all sherds in the Solazzo and Erhardt 2007 study.

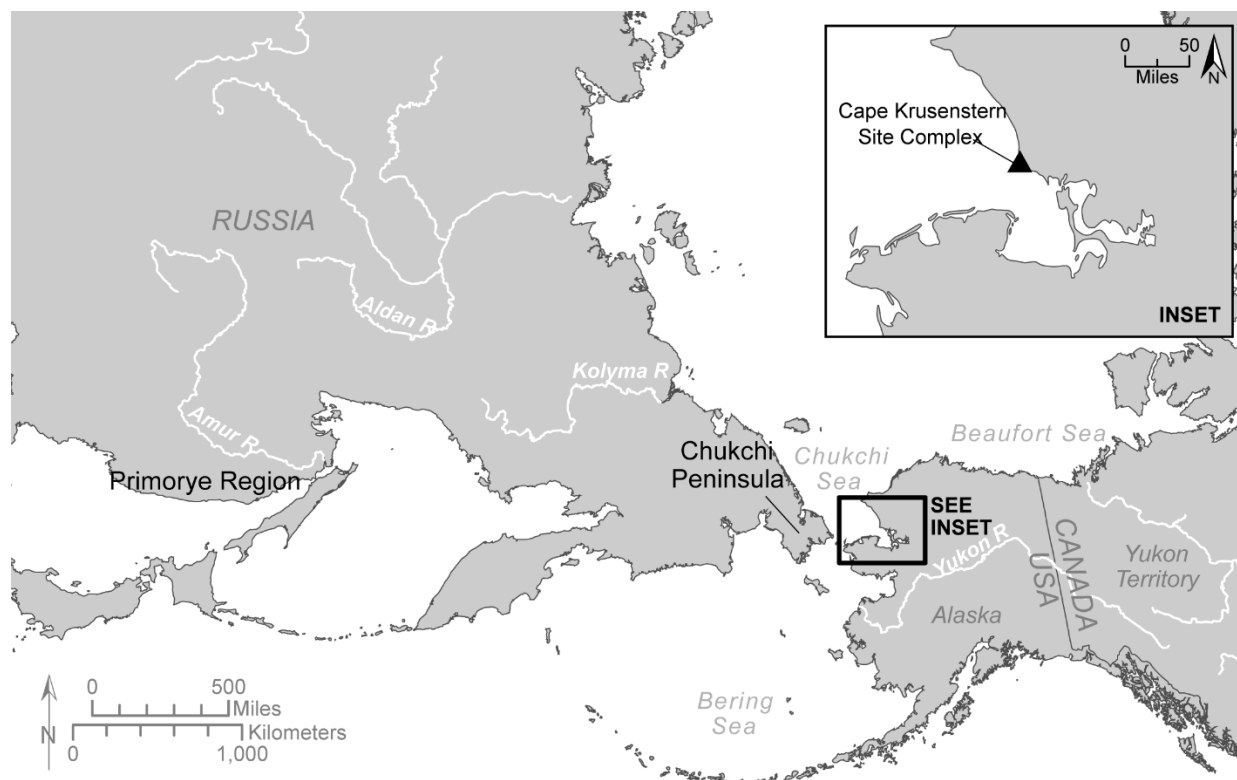


Figure 1. Regions mentioned in text where early Arctic pottery technologies are found in relationship to the Northwest Alaskan study area and site (see inset).

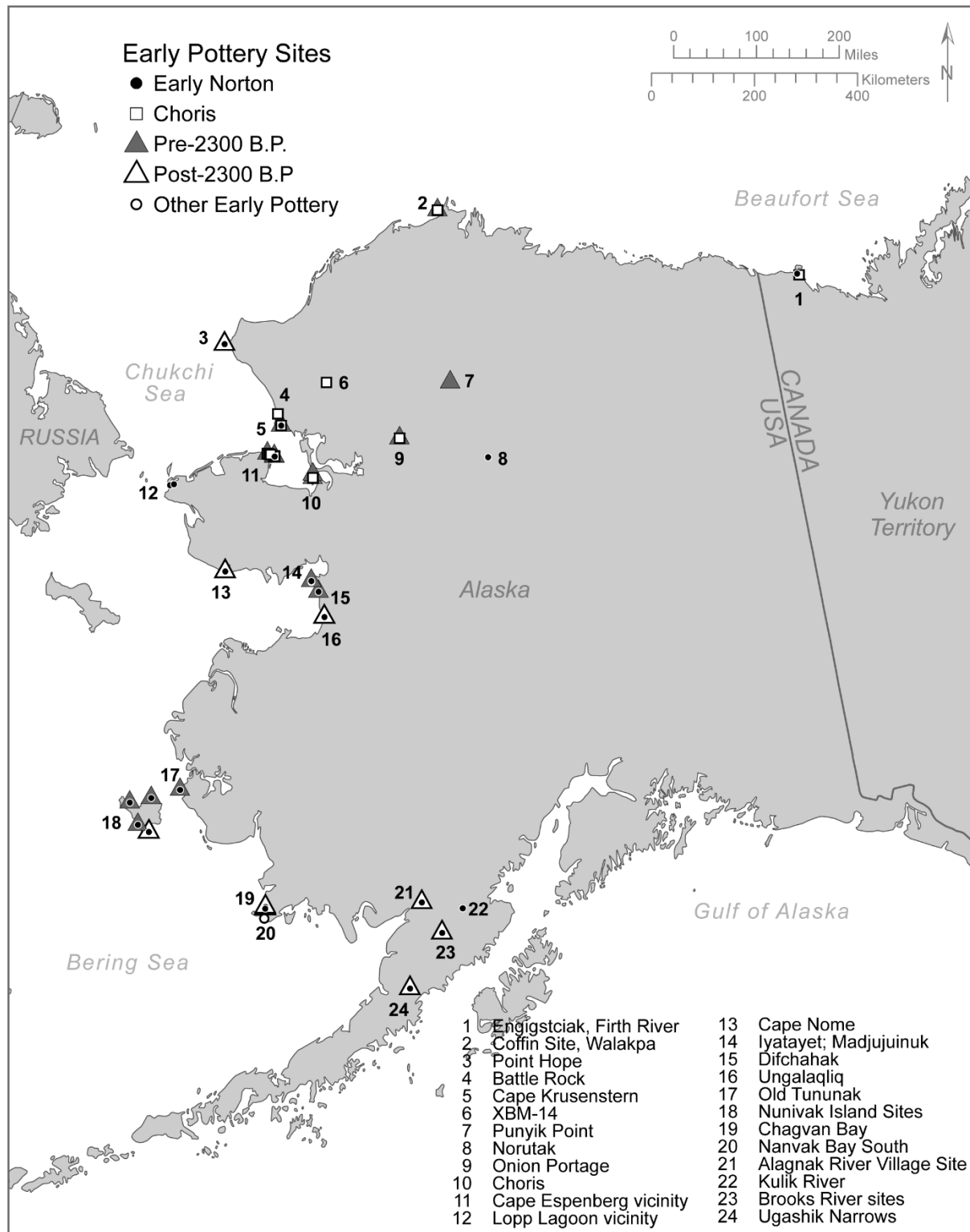


Figure 2. Early pottery sites (pre-1500 B.P.) in Alaska.

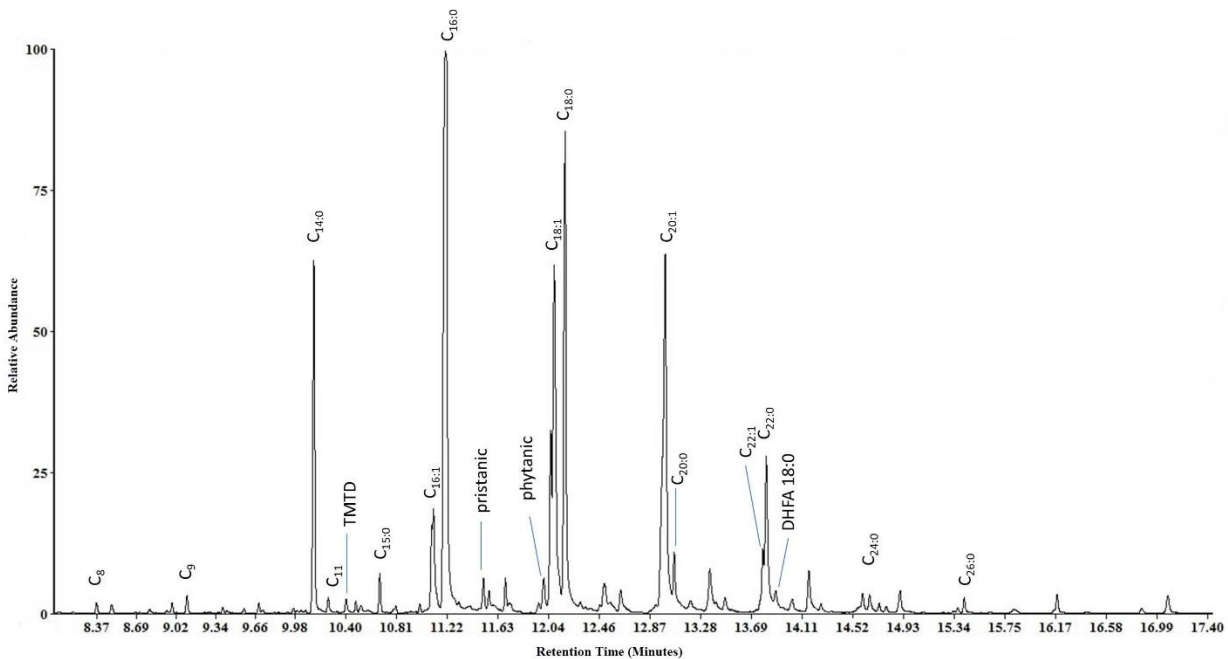


Figure 3. Total ion count chromatogram (TIC) for sample 14515b.



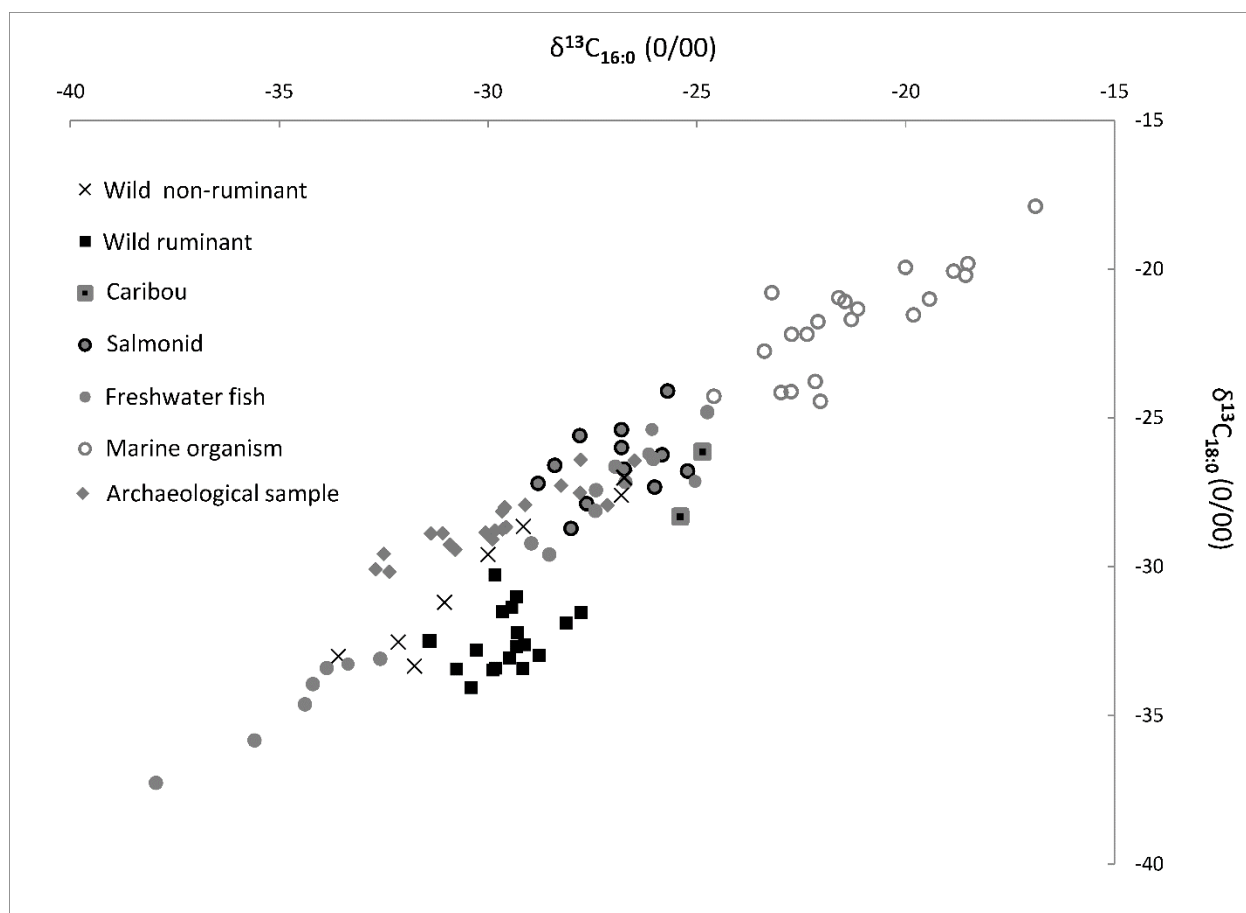


Figure 4. Compound specific  $\delta^{13}\text{C}$  values from the current project plotted with reference data from Choy et al. (2016:Table S2) and Taché and Craig (2015:Table S2).

Table 1. Early Pottery Sites in Alaska

Site	Site #	Radiocarbon Dates as Reported by	Cultural Affiliation as Reported by	Pottery Surface Treatment	Reference
		Investigator <sup>1</sup>	Investigator		
Coffin, Walakpa Bay	BAR-14	n/a	Denbigh-Choris transition	Linear stamp ("incised), Check Stamp, Plain, Chevron incised, Plain	Stanford 1971:11-12, see also Ackerman 1982; Stanford 1976
Punyik Point, Itivlik Lake Site 9	XHP-308	2600 B.P., 3660±150 B.P.	Arctic Small Tool Tradition (Denbigh)	Cord-marked	Gal 1982; Irving 1962:78.
Walakpa	BAR-13	3400 ±520 (1450±520 B.C.) to 2260±300 (310±300 B.C.)	Denbigh-Choris transition		Stanford 1971
Agulaak Site 1, locality 1	TEL-12	n/a	Norton-Near Ipiutak	Check stamp	Giddings and Anderson 1986: 162
Agulaak Site 3, locality 1	TEL-12	n/a	Norton/late Choris	Linear stamp	Giddings and Anderson 1986: 225
Battle Rock	NOA- 00078	n/a	Choris, Early Norton	Possible Linear stamp or Cord marked (difficult to discern)	Giddings and Anderson 1986:178
Cape Espenberg	KTZ-114	n/a	Choris/Norton	Linear stamp	Schaaf 1988:156

Cape Espenberg	KTZ-125	n/a	Choris/Norton	Linear stamp	Schaaf 1988:156
Cape Espenberg	KTZ-353	n/a	Choris		Tremayne 2014
Cape Espenberg	KTZ-354	n/a	Choris		Tremayne 2014
Cape Espenberg	KTZ-362	n/a	Choris		Tremayne 2014
				Linear stamp with fine twisted	
Cape Espenberg	KTZ-78	n/a	Choris	cord	Schaaf 1988: 165
				Linear stamp with fine twisted	
Cape Espenberg	KTZ-80	n/a	Choris	cord	Schaaf 1988: 165
				Linear stamp with fine twisted	
Cape Espenberg	KTZ-84	n/a	Choris	cord	Schaaf 1988: 165
				Linear stamp, Cord marked,	Giddings and Anderson 1986:
Cape Espenberg		n/a	Choris	Check stamp	226-227
Cape Espenberg	KTZ-133	2850±70 B.P.	Choris or Norton	Check stamp	Schaaf 1988: 151
		2500±90 B.P., 2285±90		Linear stamp with fine twisted	
Cape Espenberg	KTZ-98	B.P.	Choris/Norton	cord	Schaaf 1988: 164
Cape Espenberg	KTZ-109	2285±90 B.P.	Norton	Check stamp, Cord-marked	Schaaf 1988:161-162
Cape Krusenstern,					Giddings and Anderson 1986:
Beaches 36-44	NOA-2	2500±100 B.P.	Norton-Near Ipiutak	Check stamp	171

Cape Krusenstern, Beaches 44-52	NOA-2	n/a	Choris, Norton	Check stamp, Linear stamp (or possibly cord marked), Cord marked	Giddings and Anderson 1986:210, 211
					Bureau of Indian Affairs (BIA) Alaska Native Claims Settlement Office (ANCSA) 1997; Giddings and Anderson
Choris Areas	SLK-46	2635±120 B.P., 2244±133 B.P., 2646±177 B.P.	Choris	Check stamp, Linear stamp	1986:222
Choris Village	SLK-7	2635±120 B.P., 2244±133 B.P., 2646±177 B.P.	Choris	Linear stamp	BIA ANCSA 1997; Giddings and Anderson 1986:192-194.
Ipiutak	XHP-3	1970 ±100 B.P. (20 B.C.), 2070 ± 100 B.P. (120 B.C.)	Norton-Near Ipiutak	Linear stamp or Cord marked	Larsen and Rainey 1948:164; Larsen 1968:82-83
Kugzruk Site 1	TEL-149	n/a	Norton-Near Ipiutak	Check stamp, Linear stamp	Giddings and Anderson 1986: 163
N/A	XBM-14 (NR-1)	n/a	Denbigh-Choris transition	Linear stamp	Anderson 1972
Norutak 1	HUG-5	n/a	Possible Norton or Choris	Linear stamp, Plain	Clark 1974; see also Dumond 2000:8
Onion Portage	AMR-170	2370±50 B.P.	Choris		Anderson 1988: 103

Singauruk Channel				Check stamped, Possible Cord	Giddings and Anderson 1986:
Sites 1 and 2	TEL-11	n/a	Norton-Near Ipiutak	Marked	168
		2280 ±97 B.P. (330±97			
		B.C.), 2107±79 (157±79			
		B.C.),2030±99 B.P.			
Cape Nome		(80±99 B.C.)	Norton (Early)	Check stamp, Linear stamp	Bockstoce 1979: 52-56
		2330-2120 cal B.P. (for			
Difchahak	NOB-5	house w/pottery)	Norton	Check stamp	Harritt 2010
		2530±330 B.P. (580 B.C.)			
		to 2016 ±250 B.P. (66		Check stamp, Linear stamp,	Giddings 1964: 174-174,
Iyatayet	NOB-2	B.C.)	Norton	Plain	Griffin and Wilmeth 1964
Madjujuinuk					
(North Bay)	NOB-8	n/a	Norton	Check stamp, Cord marked	Giddings 1964:178
		2154±52 B.P. (204±52			
		B.C.), 2036±52 B.P.			
Ungalaqliq, Airport		(86±52 B.C.), 1603±49		Check stamp (or possibly	
Village Site	UKT-7	B.P. (A.D. 347±49)	Norton	dentate stamp)	Lutz 1970, Lutz 1972
Alagnak River				Check stamp, Diamond	
Village Site	DIL-161	2140-1300 cal B.P.	Norton	Stamp, Pigment decoration	Bundy 2007

		2255±80 B.P. (305 B.C.)			
Brooks River		to 1900±150 B.P. (A.D.	Norton Smelt Creek		
Region	XMK-51	50)	Phase	Check stamp, Diamond stamp	Dumond 1981: 132-146, 213
Chagvan Bay		2173±382 B.P. (223 B.C.)	Norton	Cord marked	Ackerman 1982:19
Chagvan Bay Bluff		1904±360 B.P., 1850±100			
Site	XHI-4	B.P.	Norton	Linear Stamp	Ackerman 1982:18
		1740±60 B.P. (A.D. 210),			
Chagvan Bay Bluff		1290±250 B.P. (A.D.			
Site	XHI-4	660)	Norton	Check Stamp	Ackerman 1982:18
Chagvan Bay,					
Southwest Alaska		n/a		Diamond stamp	Ackerman 1982
			Norton Smelt Creek		
Kulik River	XMK-48		Phase	Check stamp, Plain	Dumond 1971
		2110±95 B.P. (160±95			
		B.C.), 1885±90 B.P.			
		(A.D. 65±90), and			
		1665±80 B.P. (A.D.			Henn 1978: 46, 132; see also
Ugashik Narrows	UGA-1	285±80)	Norton	Check stamp	Ackerman 1982

Engigstciak, Firth River		n/a	Firth River Cordmarked (Choris or Norton?)	Cord marked	MacNeish 1956: see also Ackerman 1982:101-102, Griffin and Wilmeth 1964
Engigstciak, Firth River		n/a	Firth River Grooved (Norton)	Check stamp, Linear stamp	MacNeish 1956: 101-102 in Ackerman 1982
Ciguralegmiut	XCM-1	2260±80 B.P.	Norton	Check stamp	BIA ANCSA 1995 in Griffin 2002
		2580±40 B.P., 2185±50 B.P., 1900±50 B.P. Total occupation range 2600-			
Ellikarmiut, Nash Harbor	XNI-3	1900 B.P.	Norton	Check stamp	Griffin 2002
Nanvak Bay South	XHI-10	n/a		Check stamp, Cord marked	Larsen 1950:183
Penacuarmiut, Binajoaksmiut	XCM-5	2670±220 B.P., 560±100, B.P.	Norton	Check stamp, Plain	BIA ANCSA 1995 in Griffin 2002
Tanunak Site 1, Old Tununak	XNI-10	3050±270 B.P. - 2530±200 B.P.	Norton	Check stamp	Okada 1982 in Griffin 2002
		2600-1300 B.P. (150			
n/a	XNI-28	B.C.- A.D. 650)	Norton	Check stamp, Linear stamp	Nowak 1982, 1988

<sup>1</sup>It is often not clear, particularly in older publications, whether or not the dates were calibrated. They are presented here as they were by the original investigator. If dates were originally provided in B.C./A.D. format, those dates are provided in parentheses.

Table 2. List of Analyzed Ceramic Specimens and Associated Dates from the Cape Krusenstern Site Complex.

Sample Catalog No.	Ceramic Provenience	Other Info	Radiocarbon Specimen Lab No.	Conventional Radiocarbon Age (BP) <sup>1</sup>	2 Sigma Calibrated Date Range (cal BP) <sup>2</sup>	Dated Specimen Provenience Information	Material Dated	Notes on Association
CAKR 13418	Surface Scatter 2B/Hearth 2B, 1599B, Surface, PROXH071909A	Undecorated body sherd, no surface residue	OS-78589	2380±25	2486-2345	Surface Scatter 2B/Hearth 2B, 1599B, Surface, PROXH071909A	Charcoal, <i>Salicaceae</i>	
			OS-81610	2480±25	2720-2458	Activity Area 2604B Level 2, 15 cmbd, 09PROXH070308A	Charcoal, <i>Picea</i>	Date from nearby feature that is associated with location of ceramic sample
CAKR 13877b	House 1B of 09PROXH062508A, 2602B, Level 1, 0-22 cmbd, 09PROXH062709A	Undecorated body sherd, interior surface residue	OS-81679	345±25	484-314	House 1B of 09PROXH062508A 2602B, 6 cmbd, Level 1, 09PROXH062709A	Charcoal, <i>Picea</i>	
			OS-81644	910±35	918-744	House 1B OF 09PROXH062508A 2602B, 27-41 cmbd, Level 4, 09PROXH062709A	Charcoal, <i>Salicaceae</i>	
			OS-81678	650±30	670-556	House 1B of 09PROXH062508A 2602B, 60 cmbd, Level 9, 09PROXH062709A	Charcoal, <i>Salicaceae</i>	
CAKR 13884e	House 1B of 09PROXH062508A 2602B, Level 2, 10-25 cmbd, 09PROXH062709A	Undecorated body sherd, interior surface residue	OS-81679	345±25	484-314	House 1B OF 09PROXH062508A, 2602B, 6 cmbd, Level 1, 09PROXH062709A	Charcoal, <i>Picea</i>	
			OS-81644	910±35	918-744	House 1B OF 09PROXH062508A, 2602B, 27-41 cmbd,	Charcoal, <i>Salicaceae</i>	



						Level 4, 09PROXH062709A		
			OS-81678	650±30	670-556	House 1B OF 09PROXH062508A, 2602B, 60 cmbd, Level 9, 09PROXH062709A	Charcoal, <i>Salicaceae</i>	
CAKR 14026g	Surface Scatter 1B 2997B, Surface, 09PROXH071109A	Undecorated body sherd, no surface residue	OS-81682	2510±45	2747- 2435	Hearth 2B, 2992B, 5 cmBS, 09PROXXH071109A	Charcoal, <i>Salicaceae</i>	Date from nearby feature that is associated with location of ceramic sample
			OS-81654	2420±35	2700- 2350	Hearth 2B OF 09PROXH072908A, 437A, 10 cmBD, Level 1, 09GEOXH080214A	Charcoal, <i>Picea</i>	Date from nearby feature that is associated with location of ceramic sample
			OS-81648	2350±35	2487- 2315	Hearth 2B OF 09PROXH072908A, 437A, 12 cmBD, Level 1, 09GEOXH080214A	Charcoal, <i>Picea</i>	Date from nearby feature that is associated with location of ceramic sample
CAKR 14106c	Surface Scatter 2B, 2926B, Level 2, 09PROXH071008A	Undecorated body sherd, interior surface residue	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14107c	Surface Scatter 2B, 2926B, Level 2, 09PROXH071008A	Undecorated body sherd, exterior surface residue	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14109a	Surface Scatter 2B, 2926B, Level 2, 09PROXH071008A	Undecorated body sherd, interior surface residue	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14110a	Surface Scatter 2B, 2926B, Level 2, 09PROXH071008A	Undecorated body sherd, interior	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample

		surface residue						
CAKR 14112	Surface Scatter 2B, 2926B, Level 2, 09PROXH071008A	Undecorated body sherd, interior and exterior surface residues	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14113g	Surface Scatter 2B, 2926B, Level 2, 09PROXH071008A	Undecorated body sherd, interior and exterior surface residues	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14140a	Surface Scatter 2B, 2925B, Level 2, 09PROXH071008A	Undecorated body sherd, interior and exterior surface residues	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14141f	Surface Scatter 2B, 2925B, Level 2, 09PROXH071008A	Undecorated body sherd, exterior surface residue	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14142a	Surface Scatter 2B, 2925B, Level 2, 09PROXH071008A	Undecorated body sherd, interior surface residue	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14143c	Surface Scatter 2B, 2925B, Level 2, 09PROXH071008A	Undecorated body sherd, interior surface residue	OS-81680	495±25	543-505	Surface Scatter 2B, 2925B, 12cmBS, Level 1, 09PROXH071008A	Charcoal, <i>Picea</i>	Date from another unit in same feature as ceramic sample
CAKR 14514c <sup>3</sup>	Late Western Thule House 25, 34C, Disturbed Surface, 08PROXH073009A	Exfoliated surfaces, body sherd, no surface residue		770±120	925-545	Collected from area in house disturbed by previous excavation		See Giddings and Anderson 1986 for feature details

CAKR 14515b <sup>3</sup>	Late Western Thule House 27, 35C, Disturbed Surface, 08PROXH073009A	Undecorated body sherd, exterior surface residue		770±120	925-545	Collected from area in house disturbed by previous excavation	See Giddings and Anderson 1986 for feature details
CAKR 14861b	House 1A of 10GEOXH063008A 5437B, Level 4, 10PROXH070808A	Undecorated body sherd, no surface residue	OS-93763	290±35	464-158	House 1A OF 10GEOXH063008A, 5437B, 16.5 cmBD, Level 1, 10PROXXH070808A	Charcoal, <i>Picea</i>
			OS-93880	740±25	726-660	House 1A OF 10GEOXH063008A, 5437B, 88 cmBD, Level 8, 10PROXXH070808A	Wood, <i>Picea</i>
			Beta-326115	510±30	622-505	House 1A OF 10GEOXH063008A, 5437B Level 5, 10PROXXH070808A	Antler, <i>Rangifer tarandus</i>
CAKR 15110	Unidentified/Indeterminat e Feature 4B of 09PROXH062209A, 5703B, LEVEL 3, 37 N 14 E, 35 cmBD, 10PROXH071409A	Exfoliated surfaces, body sherd, no surface residue	OS-93947	305±25	458-301	Unidentified/Indetermin ate 4B OF 09PROXH062209A, 5703B, 26 cmbd, Level 2, 10PROXH071409A	Charcoal, <i>Salix</i>
			OS-93948	685±30	684-561	Unidentified/Indetermin ate 4B OF 09PROXH062209A, 5703B, 41 cmbd, Level 4, 10PROXH071409A	Charcoal, <i>Salix</i>
			OS-93934	755±25	727-667	Unidentified/Indetermin ate 4B OF 09PROXH062209A, 5703B, 66 cmbd, Level 6, 10PROXH071409A	Charcoal, <i>Salix</i>
CAKR 15146	Unidentified/Indeterminat e Feature 4B of 09PROXH062209A, 5703B, Level 5, 51 cmBD, 10PROXH071409A	Exfoliated surfaces, body sherd, no surface residue	OS-93947	305±25	458-301	Unidentified/Indetermin ate 4B OF 09PROXH062209A, 5703B, 26 cmbd, Level 2, 10PROXH071409A	Charcoal, <i>Salix</i>

			OS-93948	685±30	<i>684-561</i>	Unidentified/Indeterminate 4B OF 09PROXH062209A, 5703B, 41 cmbd, Level 4, 10PROXH071409A	Charcoal, <i>Salix</i>
			OS-93934	755±25	<i>727-667</i>	Unidentified/Indeterminate 4B OF 09PROXH062209A, 5703B, 66 cmbd, Level 6, 10PROXH071409A	Charcoal, <i>Salix</i>
CAKR 15151 <sup>3</sup>	Late Western Thule House 27, 5715B, Surface, 10PROXH072110A	Exfoliated surfaces, body sherd, interior surface residue		770±120	925-545	Collected from area in house disturbed by previous excavation	See Giddings and Anderson 1986 for feature details

<sup>1</sup>Dates calibrated using OxCal v.4.2 (Bronk Ramsey 2009), IntCal 13 (Reimer et al. 2013)

<sup>2</sup>Most closely associated date italicised

<sup>3</sup>Date ranges for these samples based on published dates for Late Western Thule House 25 (Giddings and Anderson 1986)

Table 3. Summary of Residue Results

Sample	$\delta^{13}\text{C}_{16:0}$	$\delta^{13}\text{C}_{18:0}$	4,8,12-TMTD	Phytanic	Pristanic	APAAs <sup>1</sup>	Other lipids <sup>2,3,4,5</sup>	Ratio of $\text{C}_{16:0}/\text{C}_{18:0}$	FA $\mu\text{g g}^{-1}$
13418	-31.089	-28.880		Y			SFA ( $\text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}$ ); MUFA ( $\text{C}_{16}, \text{C}_{18}$ ); DCA( $\text{C}_9\text{-C}_{11}$ )	1.90	84.9
13877b	-29.662	-28.148	Y				SFA ( $\text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA ( $\text{C}_{16}, \text{C}_{18}, \text{C}_{20}$ ); DCA ( $\text{C}_9$ )	2.51	56.0
13884e	-29.571	-28.673		Y			SFA ( $\text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA ( $\text{C}_{16}, \text{C}_{18}$ ); DCA ( $\text{C}_9$ ); hydroxy FA( $\text{C}_{16}$ )	1.61	53.6
14026g	-29.896	-29.076				$\text{C}_{18}$	SFA ( $\text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{17}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA ( $\text{C}_{18}, \text{C}_{22}$ )	1.77	58.4
14106c	-27.781	-26.400					SFA ( $\text{C}_{14}, \text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}$ ); MUFA ( $\text{C}_{18}, \text{C}_{22}$ )	1.64	79.7
14107c	-30.058	-28.856				$\text{C}_{18}$	SFA ( $\text{C}_{12}, \text{C}_{13}, \text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{17}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}$ ); MUFA( $\text{C}_{16}, \text{C}_{18}$ ); DCA ( $\text{C}_8\text{-C}_{11}$ ), DH( $\text{C}_{18}$ )	0.94	106.5
14109a	-32.363	-30.172				$\text{C}_{18}$	SFA ( $\text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA ( $\text{C}_{18}, \text{C}_{22}$ )	1.36	65.9
14110a	-28.249	-27.276				$\text{C}_{18}$	SFA ( $\text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}$ ); MUFA ( $\text{C}_{18}$ );	0.52	70.6
14112	-29.656	-28.746				$\text{C}_{18}$	SFA ( $\text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{17}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA( $\text{C}_{16}, \text{C}_{18}, \text{C}_{22}$ ), DH( $\text{C}_{18}$ )	0.86	54.5
14113g	-32.499	-29.568					SFA ( $\text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA( $\text{C}_{18}$ )	1.18	59.4
14140a	-32.694	-30.091					SFA ( $\text{C}_{14}, \text{C}_{16}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{26}$ ); MUFA ( $\text{C}_{18}, \text{C}_{22}$ )	1.05	64.3
14141f	-29.596	-28.005					SFA ( $\text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{17}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA( $\text{C}_{18}$ )	2.00	96.0
14142a	-29.112	-27.924				$\text{C}_{18}$	SFA ( $\text{C}_{12}, \text{C}_{13}, \text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{17}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}, \text{C}_{26}$ ); MUFA( $\text{C}_{16}, \text{C}_{18}$ ); DCA ( $\text{C}_8\text{-C}_{11}$ ), DH( $\text{C}_{18}$ )	1.95	65.6
14143c	-30.915	-29.255				$\text{C}_{18}$	SFA ( $\text{C}_{12}, \text{C}_{14}, \text{C}_{15}, \text{C}_{16}, \text{C}_{17}, \text{C}_{18}, \text{C}_{20}, \text{C}_{22}, \text{C}_{24}$ ); MUFA ( $\text{C}_{16}, \text{C}_{18}, \text{C}_{22}$ ); DCA ( $\text{C}_9$ ), DH( $\text{C}_{18}$ )	2.64	64.7

14514c	-27.143	-27.930	Y	Y		C <sub>18</sub> , C <sub>20</sub>	SFA (C <sub>9</sub> , C <sub>10</sub> , C <sub>11</sub> , C <sub>12</sub> , C <sub>13</sub> , C <sub>14</sub> , C <sub>15</sub> , C <sub>16</sub> , C <sub>17</sub> , C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> , C <sub>24</sub> , C <sub>26</sub> ); MUFA (C <sub>16</sub> , C <sub>18</sub> ); DCA (C <sub>7</sub> -C <sub>12</sub> ); DH(C <sub>18</sub> ), cholesterol	8.37	111.4
14515b	-27.797	-27.518	Y	Y	Y	C <sub>18</sub> , C <sub>20</sub>	SFA (C <sub>9</sub> , C <sub>10</sub> , C <sub>11</sub> , C <sub>12</sub> , C <sub>13</sub> , C <sub>14</sub> , C <sub>15</sub> , C <sub>16</sub> , C <sub>17</sub> , C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> , C <sub>24</sub> , C <sub>26</sub> ); MUFA (C <sub>16</sub> , C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> ); DCA (C <sub>8</sub> -C <sub>11</sub> ); DH(C <sub>18</sub> )	1.53	110.0
14861b	-31.365	-28.881				C <sub>18</sub>	SFA (C <sub>12</sub> , C <sub>13</sub> , C <sub>14</sub> , C <sub>15</sub> , C <sub>16</sub> , C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> , C <sub>26</sub> ); MUFA(C <sub>16</sub> , C <sub>18</sub> )	2.46	79.9
15110	-29.831	-28.784					SFA (C <sub>14</sub> , C <sub>16</sub> , C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> , C <sub>26</sub> ); MUFA (C <sub>18</sub> )	2.91	68.4
15146	-30.786	-29.433					SFA (C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> ); MUFA (C <sub>18</sub> ,)	0.55	59.1
15151	-26.492	-26.431	Y	Y	Y	C <sub>18</sub> , C <sub>20</sub>	SFA (C <sub>10</sub> , C <sub>11</sub> , C <sub>12</sub> , C <sub>13</sub> , C <sub>14</sub> , C <sub>15</sub> , C <sub>16</sub> , C <sub>17</sub> , C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> , C <sub>24</sub> , C <sub>26</sub> ); MUFA (C <sub>16</sub> , C <sub>18</sub> , C <sub>20</sub> , C <sub>22</sub> ); DCA (C <sub>8</sub> -C <sub>12</sub> ); DH(C <sub>18</sub> ), cholesterol	2.94	87.6

<sup>1</sup>APAA= $\omega$ -(*o*-alkylphenyl)alkanoic acids

<sup>2</sup>SFA = saturated fatty acids

<sup>3</sup>MUFA=monounsaturated fatty acids

<sup>4</sup>DCA= $\alpha,\omega$ -dicarboxylic acids

<sup>5</sup>DH=dihydroxy fatty acids

## **Supplemental Text 1: Residue Methods**

### *Sample preparation*

Sample preparation and CSIA extraction was performed at UC Davis by ST using methods based on those developed in similar studies (Eerkens, 2001; Evershed et al., 2002; Tushingham et al., 2013). Strict protocols were followed throughout sample preparation to avoid contamination. Prior to analysis, all artifacts were inspected for visible residue. A small (~1cm diameter) fragment of each potsherd was broken off and ~1mm of all exposed surfaces was removed with an abrasive silicon carbide/ steel dremel drill bit. Fragments were crushed into a powder using a small agate mortar and pestle and divided for CSIA and GC/MS analysis.

### **Compound specific $\delta^{13}\text{C}$ analysis**

For each sample, 200mg of crushed material was submersed in 2ml of a chloroform-methanol solvent (2:1, v/v), vortexed, sonicated for 20 minutes, and then centrifuged to separate the solvent mixture, now containing lipids, from the fine clay particles. The lipid extract was transferred to a second tube and evaporated under a gentle stream of nitrogen. Lipid extracts were derivatized by adding 100 $\mu\text{l}$  of methanolic HCl to the dried lipids, and heating at 60°C for 1 hour. Derivatized lipids were extracted with hexane and transferred into 2ml GC vials for

compound-specific  $^{13}\text{C}$  isotope analysis (CSIA) of individual fatty acids using GC combustion isotope ratio mass spectrometry (GC-C-IRMS).

Compound specific stable isotope analysis was performed at the UC Davis Stable Isotope Facility (<http://stableisotopefacility.ucdavis.edu/>). Compounds were analyzed on a Trace GC Ultra gas chromatograph coupled to a Delta V Advantage isotope ratio mass spectrometer through a GC-C-III interface. Samples were injected, splitless, on a VF-5ms column (30m x 0.25mm ID, 0.25  $\mu\text{m}$  film thickness). Once separated, FAMES were quantitatively converted to  $\text{CO}_2$  in a  $\text{CuO/NiO/Pt}$  oxidation reactor at  $950^\circ\text{C}$ , dried, and introduced to the IRMS. Corrections to provisional IRMS values were made based on working standards composed of FAMES calibrated against NIST standard reference materials. The UC Davis Stable Isotope Facility reports that their long-term estimate of measurement error for CSIA of FAMES is generally better than  $\pm 0.5\%$ .

The  $\delta^{13}\text{C}$  values for FAME samples are expressed in permil as ratios of  $^{13}\text{C}$  to  $^{12}\text{C}$  relative to the ratio for the standard reference, V-PDB. The  $\delta^{13}\text{C}$  values were calculated as follows: ( $\text{‰}$ )  
$$= (\text{R sample} - \text{R standard} / \text{R standard}) \times 1000$$
, where R is equal to the ratio of the heavy to the light isotope ( $^{13}\text{C}/^{12}\text{C}$ ) in the sample compared with that of the standard. Final  $\delta^{13}\text{C}$  values for sample FAMES were corrected for the isotopic contribution of methanol, incorporated during fatty acid derivatization, using a mass balance equation (Regert 2011:196).

#### *GC/TOF-MS*

GC/TOF-MS analysis was conducted by the Laboratory for Cellular Metabolism and Engineering Analytics facility at Washington State University. Lipids were extracted using a



modified Bligh and Dyer technique similar to that employed in Buonasera et al. (2015). Crushed sherds (~1g) were extracted by sonication in 10ml of chloroform-methanol-water at a ratio of 1:2:0.8 (v/v/v) for 15min. After a 10min rest, the sonication was repeated. After a brief centrifugation, the solvent was removed, and the crushed pottery was washed with 2ml of the above solvent mixture, and the washing fraction combined with the extracts. For phase separation, 3ml chloroform and 3ml water were added. The chloroform phase was transferred to a new tube. The remaining aqueous phase was re-extracted with additional 3ml of chloroform. The combined chloroform extracts were dried under a gentle stream of nitrogen. The extracted lipids were derivatized with 3ml 1.25M HCl for 60 min at 60°C. After cooling, the solution was neutralized with saturated sodium bicarbonate solution and the derivatized fatty acids extracted with hexane. The hexane phase was dried under a stream of nitrogen, and the dry residue dissolved in chloroform and analyzed as below.

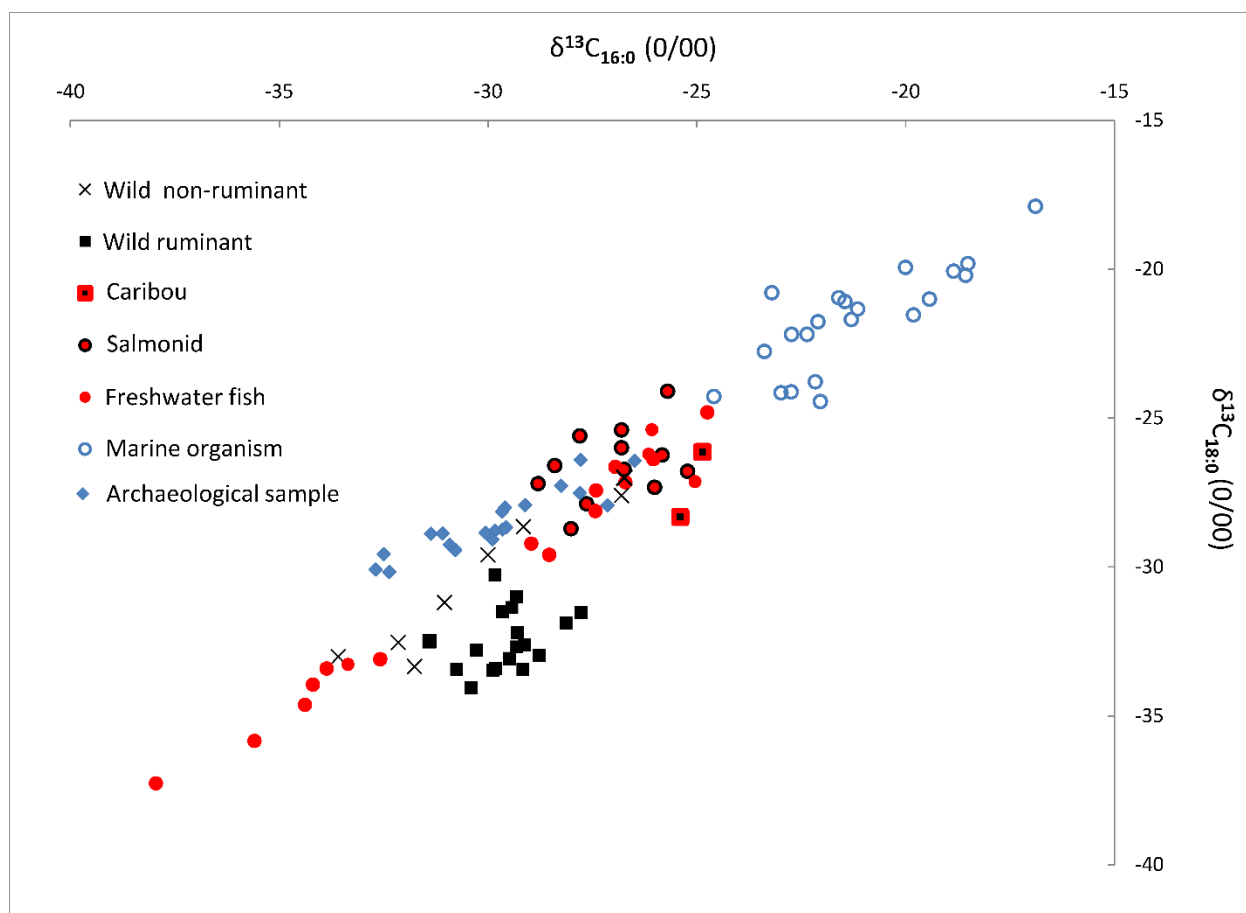
Derivatized lipid extracts were analyzed on an Agilent 7890A gas chromatograph coupled with a Pegasus 4D time-of-flight mass spectrometer (LECO), and signals were integrated using ChromaTOF software (LECO) and NIST library. The GC was fitted with an Rxi-5Sil® column (Restek), 30m x 0.25mm, 0.25µm df column, the carrier gas was He at a constant flow of 1ml min<sup>-1</sup>, and the inlet temperature was set to 250°C. Samples (1µl) were injected by a Gerstel MPS2 autosampler and split 1:15. After a 1 minute isothermal hold at 50°C, the temperature was ramped to 330°C at 20°C per minute, with a 5 minute final isothermal hold at 330°C. Mass spectra were collected at 17 spectra s<sup>-1</sup>.

Total ion count data for all samples were analyzed as .cdf files by TB with AMDIS 32, version 2.71. Lipid compounds were identified by comparing mass spectra for the samples to those in the NIST Standard Reference Database and to standard reference compounds (Supelco

SP-37 FAME mixture) run the same instrument. Detection of  $\omega$ -(*o*-alkylylphenyl)alkanoic acids 18, 20, and 22 carbons long was accomplished by analyzing mass spectra for selected ions. The compounds were identified by the presence of a dominant ion at  $m/z$  105 along with  $M^+$  ions for C18 (290), C20 (318) and C22 (346)  $\omega$ -(*o*-alkylylphenyl)alkanoic acids (Evershed et al., 2008; Hansel et al., 2004; Heron et al., 2010). The dominant ion at  $m/z$  105 represents a dialkyl benzene fragment,  $C_8H_9^+$ , common to all  $\omega$ -(*o*-alkylylphenyl)alkanoic acids (Michael, 1966).

Fatty acid concentrations were calculated from internal standard ( $C_{12:0}$ ) and fatty acid peak areas reported in the  $\delta^{13}C$  compound specific stable isotope analysis. Fatty acid concentrations could not be calculated from GC-TOF/MS data because internal standards were not added to these samples.

**Supplemental Figure 1:** Color version of Figure 4 (Compound specific  $\delta^{13}\text{C}$  values from the current project plotted with reference data from Choy et al. (2016:Table S2) and Taché and Craig (2015:Table S2)).



**Supplemental Table 1.** Compound specific  $\delta^{13}\text{C}$  values for palmitic acid ( $\text{C}_{16:0}$ ) and stearic acid ( $\text{C}_{18:0}$ ) plotted in Figure 4.

Category	Species	Sample	Location	$\delta^{13}\text{C}_{16:0}$	$\delta^{13}\text{C}_{18:0}$	$\Delta^{13}\text{C}$	Reference
Wild non-ruminant	<i>Ursus americanus</i> (Black bear)	bone	Canada	-26.73	-27.00	-0.27	Taché & Craig 2015, Table S2
Wild non-ruminant	<i>Castor canadensis</i> (Beaver)	soft tissue	Canada	-31.04	-31.20	-0.16	Taché & Craig 2015, Table S2
Wild non-ruminant	<i>Lepus americanus</i> (Snowshoe hare)	soft tissue	Canada	-32.15	-32.54	-0.39	Taché & Craig 2015, Table S2
Wild non-ruminant	<i>Procyon lotor</i> (Raccoon)	soft tissue	Canada	-29.15	-28.65	0.50	Taché & Craig 2015, Table S2
Wild non-ruminant	<i>Ondatra zibethicus</i> (Muskrat)	soft tissue	Canada	-33.58	-33.02	0.56	Taché & Craig 2015, Table S2
Wild non-ruminant	<i>Lontra Canadensis</i> (Otter)	soft tissue	Canada	-31.76	-33.34	-1.58	Taché & Craig 2015, Table S2
Wild non-ruminant	<i>Lepus americanus</i> (Snowshoe hare)	muscle tissue	Interior Alaska	-30.0	-29.6	0.4	Choy et al. 2016, Table S2
Wild non-ruminant	<i>Sciurus vulgaris</i> (Red squirrel)	muscle tissue	Interior Alaska	-26.8	-27.6	-0.8	Choy et al. 2016, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-28.13	-31.89	-3.76	Taché & Craig 2015, Table S2

Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-27.77	-31.54	-3.77	Taché & Craig 2015, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-28.78	-32.98	-4.20	Taché & Craig 2015, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-30.41	-34.06	-3.65	Taché & Craig 2015, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-29.49	-33.08	-3.59	Taché & Craig 2015, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-29.16	-33.43	-4.27	Taché & Craig 2015, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-30.75	-33.44	-2.69	Taché & Craig 2015, Table S2

Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-29.88	-33.47	-3.59	Taché & Craig 2015, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-29.31	-32.69	-3.38	Taché & Craig 2015, Table S2
Wild ruminant	<i>Cervus elaphus</i> (Red deer)	bone	Poland	-29.82	-33.41	-3.59	Taché & Craig 2015, Table S2
Wild ruminant	<i>Alces alces</i> (Moose)	bone	Canada	-29.12	-32.62	-3.50	Taché & Craig 2015, Table S2

Wild ruminant	<i>Alces alces</i> (Moose)	bone	Canada	-30.27	-32.80	-2.53	Taché & Craig 2015, Table S2
Wild ruminant	<i>Alces alces</i> (Moose)	soft tissue	Canada	-29.30	-32.21	-2.91	Taché & Craig 2015, Table S2
Wild ruminant	<i>Alces alces</i> (Moose)	soft tissue	Canada	-29.43	-31.37	-1.94	Taché & Craig 2015, Table S2
Wild ruminant	<i>Alces alces</i> (Moose)	muscle tissue	Interior Alaska	-31.4	-32.5	-1.1	Choy et al. 2016, Table S2
Wild ruminant	<i>Odocoileus virginianus</i> (White-tailed deer)	bone	Canada	-29.32	-31.01	-1.69	Taché & Craig 2015, Table S2
Wild ruminant	<i>Odocoileus virginianus</i> (White-tailed deer)	soft tissue	Canada	-29.65	-31.51	-1.86	Taché & Craig 2015, Table S2
Wild ruminant	<i>Odocoileus virginianus</i> (White-tailed deer)	soft tissue	Canada	-29.83	-30.28	-0.45	Taché & Craig 2015, Table S2
Wild ruminant	<i>Rangifer tarandus</i> (Caribou)	bone	Canada	-25.40	-28.32	-2.92	Taché & Craig 2015, Table S2
Wild ruminant	<i>Rangifer tarandus</i> (Caribou)	soft tissue	Canada	-24.87	-26.14	-1.27	Taché & Craig 2015, Table S2
Salmonid (Anadromous)	<i>Oncorhynchus kisutch</i> (Coho Salmon)	muscle tissue	Interior Alaska	-28.8	-27.2	1.6	Choy et al. 2016, Table S2
Salmonid (Anadromous)	<i>Oncorhynchus kisutch</i> (Coho Salmon)	muscle tissue	Interior Alaska	-28.4	-26.6	1.8	Choy et al. 2016, Table S2

Salmonid (Anadromous)	<i>Oncorhynchus kisutch</i> (Coho Salmon)	muscle tissue	Interior Alaska	-27.8	-25.6	2.2	Choy et al. 2016, Table S2
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Salmonid (Anadromous)	<i>Oncorhynchus keta</i> (Chum Salmon)	muscle tissue	Interior Alaska	-26.8	-26.0	0.8	Choy et al. 2016, Table S2
Salmonid (Anadromous)	<i>Oncorhynchus keta</i> (Chum Salmon)	muscle tissue	Interior Alaska	-26.8	-25.4	1.4	Choy et al. 2016, Table S2
Salmonid (Anadromous)	<i>Oncorhynchus keta</i> (Chum Salmon)	muscle tissue	Interior Alaska	-25.7	-24.1	1.6	Choy et al. 2016, Table S2
Salmonid (Freshwater)	Salmonidae sp. (Salmon)	charred deposit	Japan	-28.01	-28.72	-0.71	Taché & Craig 2015, Table S2
Salmonid (Freshwater)	Salmonidae sp. (Salmon)	charred deposit	Japan	-25.22	-26.79	-1.57	Taché & Craig 2015, Table S2
Salmonid (Freshwater)	Salmonidae sp. (Salmon)	charred deposit	Japan	-26.01	-27.33	-1.32	Taché & Craig 2015, Table S2
Salmonid (Freshwater)	Salmonidae sp. (Trout)	charred deposit	Japan	-26.74	-26.72	0.02	Taché & Craig 2015, Table S2
Salmonid (Freshwater)	Salmonidae sp. (Trout)	charred deposit	Japan	-27.64	-27.88	-0.24	Taché & Craig 2015, Table S2

Salmonid (Freshwater)	Salmonidae sp. (Trout)	charred deposit	Japan	-25.83	-26.24	-0.41	Taché & Craig 2015, Table S2
Freshwater	<i>Rhynchocypris lagowskii</i> (Amur minnow)	charred deposit	Japan	-27.43	-28.13	-0.70	Taché & Craig 2015, Table S2
Freshwater	<i>Pseudorasbora parva</i> (Topmouth gudgeon)	charred deposit	Japan	-26.95	-26.64	0.31	Taché & Craig 2015, Table S2
Freshwater	<i>Anguilla anguilla</i> (Eel)	soft tissue	Denmark	-28.96	-29.22	-0.26	Taché & Craig 2015, Table S2
Freshwater	<i>Esox lucius</i> (Pike)	soft tissue	Denmark	-35.59	-35.84	-0.25	Taché & Craig 2015, Table S2
Freshwater	<i>Tinca tinca</i> (Tench)	soft tissue	Denmark	-28.53	-29.6	-1.07	Taché & Craig 2015, Table S2
Freshwater	<i>Tinca tinca</i> (Tench)	soft tissue	Denmark	-25.04	-27.14	-2.10	Taché & Craig 2015, Table S2
Freshwater	<i>Tinca tinca</i> (Tench)	soft tissue	Denmark	-37.95	-37.27	0.68	Taché & Craig 2015, Table S2
Freshwater	<i>Ictalurus punctatus</i> (Channel catfish)	soft tissue	Canada	-27.41	-27.43	-0.02	Taché & Craig 2015, Table S2
Freshwater	<i>Ictalurus punctatus</i> (Channel catfish)	soft tissue	Canada	-26.07	-25.39	0.68	Taché & Craig 2015, Table S2
Freshwater	<i>Ictalurus punctatus</i> (Channel catfish)	soft tissue	Canada	-26.71	-27.16	-0.45	Taché & Craig 2015, Table S2



Freshwater	<i>Ictalurus punctatus</i> (Channel catfish)	soft tissue	Canada	-26.15	-26.21	-0.06	Taché & Craig 2015, Table S2
Freshwater	<i>Ictalurus punctatus</i> (Channel catfish)	soft tissue	Canada	-24.75	-24.81	-0.06	Taché & Craig 2015, Table S2
Freshwater	<i>Ictalurus punctatus</i> (Channel catfish)	soft tissue	Canada	-26.04	-26.38	-0.34	Taché & Craig 2015, Table S2
Freshwater	<i>Microgadus tomcod</i> (Tomcod)	soft tissue	Canada	-34.38	-34.63	-0.25	Taché & Craig 2015, Table S2
Freshwater	<i>Microgadus tomcod</i> (Tomcod)	soft tissue	Canada	-34.19	-33.95	0.24	Taché & Craig 2015, Table S2
Freshwater	<i>Microgadus tomcod</i> (Tomcod)	soft tissue	Canada	-33.86	-33.41	0.45	Taché & Craig 2015, Table S2
Freshwater	<i>Microgadus tomcod</i> (Tomcod)	soft tissue	Canada	-33.35	-33.28	0.07	Taché & Craig 2015, Table S2
Freshwater	<i>Microgadus tomcod</i> (Tomcod)	soft tissue	Canada	-32.58	-33.10	-0.52	Taché & Craig 2015, Table S2
Marine Organism	<i>Gymnocranius euanus</i> (Sea bream)	flesh	Japan	-22.1	-21.77	0.33	Taché & Craig 2015, Table S2
Marine Organism	<i>Gymnocranius euanus</i> (Sea bream)	flesh	Japan	-22.36	-22.19	0.17	Taché & Craig 2015, Table S2
Marine Organism	Sebastes sp. (Rockfish)	flesh	Japan	-23.38	-22.76	0.62	Taché & Craig 2015, Table S2

Marine Organism	<i>Mugil cephalus</i> (Flathead mullet)	flesh	Japan	-21.6	-20.97	0.63	Taché & Craig 2015, Table S2
Marine Organism	<i>Genyonemus lineatus</i> (Croaker)	flesh	Japan	-21.45	-21.09	0.36	Taché & Craig 2015, Table S2
Marine Organism	<i>Myoxocephalus scorpius</i> (Bull trout)	flesh	Denmark	-16.89	-17.89	-1.00	Taché & Craig 2015, Table S2
Marine Organism	<i>Gadus morhua</i> (Atlantic cod)	flesh	Denmark	-22.73	-22.19	0.54	Taché & Craig 2015, Table S2
Marine Organism	<i>Gadus morhua</i> (Atlantic cod)	flesh	Denmark	-22.74	-24.12	-1.38	Taché & Craig 2015, Table S2
Marine Organism	<i>Gadus morhua</i> (Atlantic cod)	flesh	Denmark	-22.04	-24.45	-2.41	Taché & Craig 2015, Table S2
Marine Organism	<i>Zoarces viviparus</i> (Eelpout)	flesh	Denmark	-19.43	-21.01	-1.58	Taché & Craig 2015, Table S2
Marine Organism	<i>Zoarces viviparus</i> (Eelpout)	flesh	Denmark	-21.15	-21.34	-0.19	Taché & Craig 2015, Table S2
Marine Organism	<i>Platichthys flesus</i> (European flounder)	flesh	Denmark	-18.51	-19.82	-1.31	Taché & Craig 2015, Table S2
Marine Organism	<i>Pleuronectes platessa</i> (Plaice)	flesh	Denmark	-19.81	-21.54	-1.73	Taché & Craig 2015, Table S2
Marine Organism	<i>Pleuronectes platessa</i> (Plaice)	flesh	Denmark	-18.85	-20.07	-1.22	Taché & Craig 2015, Table S2

Marine Organism	<i>Phoca largha</i> (Spotted seal)	blubber	Denmark	-20.00	-19.95	0.05	Taché & Craig 2015, Table S2
Marine Organism	<i>Phoca largha</i> (Spotted seal)	blubber	Denmark	-12.80	-14.26	-1.46	Taché & Craig 2015, Table S2
Marine Organism	<i>Phoca vitulina</i> (Harbour seal)	blubber	Germany	-18.56	-20.21	-1.65	Taché & Craig 2015, Table S2
Marine Organism	<i>Gadus morhua</i> (Atlantic cod)	soft tissue	Germany	-21.30	-21.70	-0.40	Taché & Craig 2015, Table S2
Marine Organism	<i>Clupea harengus</i> (Atlantic herring)	soft tissue	Germany	-23.20	-20.8	2.40	Taché & Craig 2015, Table S2
Marine Organism	Pinnipedia sp. (Seal)	bone	Canada	-22.98	-24.15	-1.17	Taché & Craig 2015, Table S2
Marine Organism	Pinnipedia sp. (Seal)	bone	Canada	-22.16	-23.78	-1.62	Taché & Craig 2015, Table S2
Marine Organism	Pinnipedia sp. (Seal)	bone	Canada	-24.59	-24.28	0.31	Taché & Craig 2015, Table S2
Archaeological Sample-pottery	N/A	15151	Northwestern Alaska	-26.492	-26.431	0.061	Current study
Archaeological Sample-pottery	N/A	14514c	Northwestern Alaska	-27.143	-27.93	-0.787	Current study
Archaeological Sample-pottery	N/A	14106c	Northwestern Alaska	-27.781	-26.400	1.381	Current study

Archaeological Sample-pottery	N/A	14515b	Northwestern Alaska	-27.797	-27.518	0.279	Current study
Archaeological Sample-pottery	N/A	14110a	Northwestern Alaska	-28.249	-27.276	0.973	Current study
Archaeological Sample-pottery	N/A	14142a	Northwestern Alaska	-29.112	-27.924	1.188	Current study
Archaeological Sample-pottery	N/A	13884e	Northwestern Alaska	-29.571	-28.673	0.898	Current study
Archaeological Sample-pottery	N/A	14141f	Northwestern Alaska	-29.596	-28.005	1.591	Current study
Archaeological Sample-pottery	N/A	14112	Northwestern Alaska	-29.656	-28.746	0.910	Current study
Archaeological Sample-pottery	N/A	13877b	Northwestern Alaska	-29.662	-28.148	1.514	Current study
Archaeological Sample-pottery	N/A	15110	Northwestern Alaska	-29.831	-28.784	1.047	Current study
Archaeological Sample-pottery	N/A	14026g	Northwestern Alaska	-29.896	-29.076	0.820	Current study
Archaeological Sample-pottery	N/A	14107c	Northwestern Alaska	-30.058	-28.856	1.202	Current study
Archaeological Sample-pottery	N/A	15146	Northwestern Alaska	-30.786	-29.433	1.353	Current study

Archaeological Sample-pottery	N/A	14143c	Northwestern Alaska	-30.915	-29.255	1.660	Current study
Archaeological Sample-pottery	N/A	13418	Northwestern Alaska	-31.089	-28.88	2.209	Current study
Archaeological Sample-pottery	N/A	14861b	Northwestern Alaska	-31.365	-28.881	2.484	Current study
Archaeological Sample-pottery	N/A	14109a	Northwestern Alaska	-32.363	-30.172	2.191	Current study
Archaeological Sample-pottery	N/A	14113g	Northwestern Alaska	-32.499	-29.568	2.931	Current study
Archaeological Sample-pottery	N/A	14140a	Northwestern Alaska	-32.694	-30.091	2.603	Current study

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